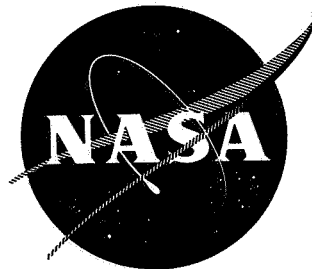


N169-27959  
NASA CR-72545

K for #

T-212898



NASA CR-72545

SMALL QUANTITY PRODUCTION OF COMPLEX CHROMIUM ALLOY SHEET  
(Cr-7Mo-2Ta-0.09C-0.1Y)

by

E. R. Slaughter, J. R. Hughes and W. F. Moore  
General Electric Company

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center

Contract NAS 3-9417

John P. Merutka, Project Manager

**CASE FILE  
COPY**

### NOTICE

This report was prepared as an account of Government-sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately-owned rights; or
- B.) Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA or employee of such contractor prepares, disseminates, or provides access to any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report should be referred to

National Aeronautics and Space Administration  
Scientific and Technical Information Facility  
P.O. Box 33  
College Park, Md. 20740

FINAL REPORT

SMALL QUANTITY PRODUCTION OF COMPLEX CHROMIUM ALLOY SHEET  
(Cr-7Mo-2Ta-0.09C-0.1Y)

by

E. R. Slaughter, J. R. Hughes and W. F. Moore

Lamp Metals and Components Department  
General Electric Company  
Cleveland, Ohio

and

Research and Development Center  
General Electric Company  
Schenectady, New York

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

December 1968

Contract NAS 3-9417

NASA Lewis Research Center  
Cleveland, Ohio

William D. Klopp, Research Advisor  
John P. Merutka, Project Manager  
Materials and Structures Division





## FOREWORD

This is to acknowledge the contribution of L. J. Goetz in the initial planning of the project and the conduct of the early phases of the rolling development.



## TABLE OF CONTENTS

	Page
INTRODUCTION. . . . .	1
MELTING AND CASTING . . . . .	3
General Approach to the Melting Problem. . . . .	3
Equipment. . . . .	3
Melt Stock . . . . .	4
Preparation of the Charge. . . . .	4
Melting Procedure. . . . .	10
Ingot Quality. . . . .	10
EXTRUSION . . . . .	19
Preparation of Ingots for Extrusion. . . . .	19
Canning. . . . .	19
Extrusion. . . . .	20
Discussion of Extrusion Data . . . . .	25
ROLLING AND ANNEALING . . . . .	29
Goals. . . . .	29
Annealing of the Extrusions. . . . .	29
Jacketing. . . . .	30
Development of Rolling and Annealing Processes . . . . .	40
Rolling and Annealing of the 1/16" Product . . . . .	52
Rolling and Annealing of the 1/4" Product. . . . .	52
Processing of Other Products . . . . .	52

## TABLE OF CONTENTS (Continued)

	Page
EVALUATION OF PRODUCTS . . . . .	53
Mechanical Properties . . . . .	53
Dimensions and Finish . . . . .	53
Cracks . . . . .	53
Structure . . . . .	53
Composition . . . . .	57
Yield . . . . .	57
APPENDIX A . . . . .	58
APPENDIX B . . . . .	62
APPENDIX C . . . . .	63
APPENDIX D . . . . .	65
REFERENCES . . . . .	67
DISTRIBUTION LIST. . . . .	69

## LIST OF TABLES

	Page
I. Chemistry of "Elchrome HP" Chromium . . . . .	5
II. Tantalum Chips. . . . .	5
III. Molybdenum. . . . .	6
IV. Nuclear Grade Yttrium Sponge. . . . .	7
V. Nuclear Grade Lanthanum Ingot . . . . .	8
VI. Spectroscopic Graphite AGKSP. . . . .	9
VII. Composition of Ingots and Products. . . . .	14
VIII. Extrusion Data. . . . .	22
IX. Tensile Properties of Extrusions and Sheet. . . . .	31
X. Ductile to Brittle Bend Transition Temperatures of Strip After Processing . . . . .	47



## LIST OF ILLUSTRATIONS

	Page
1. Typical 100 Pound Ingot. . . . .	11
2. Section of 100 Pound Ingot . . . . .	12
3. Schematic Diagram of Typical Extrusion Can and Billet Assembly. . . . .	21
4. Extrusion Constant . . . . .	26
5. Ultimate Tensile Strength of Extrusions and Sheet. . . . .	33
6. Yield Strength of Extrusions and Sheet . . . . .	34
7. Ductility of Extrusions and Sheet. . . . .	35
8. Exploded View of Pack. . . . .	36
9. Recrystallization Temperature. . . . .	41
10. Rolling and Annealing Processes. . . . .	42
11. Rolling and Annealing Processes. . . . .	43
12. Rolling and Annealing Processes. . . . .	44
13. Rolling and Annealing Processes. . . . .	45
14. Ductile to Brittle Bend Transition Temperature . . . . .	51
15. Structure of Annealed Extrusion from 100 Pound Heat. . . . .	54
16. Structure of 1/4" Plate. . . . .	55
17. Structure of 1/16" Sheet as Fabricated . . . . .	56
18. Structure of 1/16" Sheet Annealed at 2462°F (1623°K) . . . . .	56





# ABSTRACT

Eighty five pounds (39 kg) of a chromium-7% molybdenum-2% tantalum-0.09% carbon-0.1% yttrium plus lanthanum alloy were produced in various forms, principally sheet. The alloy was induction melted, cast, extruded, rolled, and annealed.



## SUMMARY

The primary purpose of this program was to provide eighty five pounds (39 kg) of a chromium-molybdenum-tantalum-carbon-yttrium-lanthanum alloy in various forms, principally sheet, for use in other studies. The alloy was induction melted, cast, extruded, rolled, and annealed.

Control of the composition during melting was complicated by the reaction of the most active alloying elements, yttrium and lanthanum, with the impurities in the melt and with the yttria-stabilized zirconia crucible. Primary solidification shrinkage, porosity and cracking reduced the yield of sound ingot to about 50% of the amount melted.

Small billets, 2-1/16" (5.24 cm) diameter, were extruded successfully into rectangular bars at reduction ratios of 3.5:1 to 4.9:1 at temperatures in the range of 2678°F (1740°K) to 2863°F (1845°K). However, 5-1/16" (12.85 cm) diameter billets tore when extruded into rectangular bars under similar conditions of reduction ratio and temperature. No completely satisfactory conditions for extruding the large billets were found; poor yields were obtained.

The rolling of the extruded bar to sheet was an involved process. Jacketing was required to protect the chromium alloy from contamination. Differences in the strength of the chromium alloy and the jacket at rolling temperatures and the differential thermal contraction between them on cooling tended to cause cracks. The chromium alloy was ductile enough for rolling in a limited range of temperature. As in the case of the extrusion process, a rolling process that was successful for small heats (extrusions) caused cracking for the larger heats (extrusions). A satisfactory process for rolling the large extrusions to sheet consisted of warm rolling at 2372°F (1573°K) with intermediate annealing treatments at 2462°F (1623°K) and 2192°F (1473°K).

The fabrication process affected the transition temperature; seemingly minor changes in fabrication resulted in changes in the transition temperature of more than 700°F (388°K).

The transition temperature of the 1/16" sheet as fabricated was about 564°F (569°K). Annealing at 2462°F (1623°K) increased the transition temperature to more than 1112°F (873°K), but increased the short time elevated temperature tensile strength significantly.



## INTRODUCTION

This report covers work that was a sequel to previous work (Reference 1). Both programs were similar in their purpose, scope and techniques.

The primary purpose of both was to provide chromium alloy in various mill forms to be used in developmental work by NASA and its contractors. In both cases, a principal use was for coating development.

The scope of both programs included the production of modest quantities of mill forms of chromium alloys by induction melting, casting, extruding, rolling and annealing and the developmental effort necessary for finding adequate melting and fabrication techniques. It was intended that the techniques which were satisfactory if not optimum in the first program be used as much as possible in the present work. A major difference between the programs was the composition of the alloys. The previous work involved a chromium-tungsten-yttrium alloy, while the present effort concerned a stronger and more complex chromium-molybdenum-tantalum-carbon-yttrium-lanthanum alloy. Melting and extruding were done at the Research and Development Center, and rolling and associated operations were done at Lamp Metals and Components Department.



## MELTING AND CASTING

General Approach to the Melting Problem - The characteristics of chromium-base alloys which must be considered in melting and casting these materials have been discussed previously,<sup>(1)</sup> and these characteristics were assumed to be valid for the present program.

The complexity of the alloy to be melted was such that the program was conducted in two phases. Phase I involved optimization of previously developed melting techniques, and Phase II consisted of the melting and casting of four 100 pound (45.36 kg) ingots necessary to produce the desired quantity of finished mill product.

It was felt that consistency from heat to heat in recovery of the specified quantity of yttrium plus lanthanum in this alloy would be difficult. Therefore, provision was made for making five 15 pound (6.8 kg) heats and one 100 pound (45.36 kg) heat with the purpose of optimizing the melting technique before proceeding with the production of the required four 100 pound (45.36 kg) ingots.

Two 15 pound (6.8 kg) heats were made using different melting techniques. These two techniques were evaluated and the better one used on the next series of three 15 pound (6.8 kg) heats to check for consistency. After evaluating all five 15 pound (6.8 kg) heats, a technique was determined for use on the Phase I 100 pound (45.36 kg) heat. This technique proved to be successful, and it was used on the remaining four 100 pound (45.36 kg) heats.

Equipment - Melting and casting operations were carried out in two vacuum induction melting furnaces with capacities for melting 50 and 200 pounds (22.68 kg and 90.72 kg) of steel respectively and tilt-pouring within their chambers. The common power source for both furnace chambers was a 200 KW, 1920 Hz motor generator set. The lowest pressure attainable in these chambers was 15 microns to 30 microns ( $2 \text{ N/m}^2$  to  $4 \text{ N/m}^2$ ) of mercury.

Crucibles were made of zirconia, stabilized with 8% yttria, and were of sizes to contain 15 pound and 100 pound (6.8 kg and 45.36 kg) melts. Pouring basins and spouts were made of lime-stabilized zirconia.

Molds consisted of uniform density, coarse-grained lime-stabilized zirconia tubes. Tube size for the 100 pound (45.36 kg) molds was 5" (12.7 cm) inner diameter (ID) x 5-3/4" (14.6 cm) outer diameter (OD) x 20" (50.8 cm) long. The 15 pound (6.8 kg) mold tube consisted of an ingot section 2-1/4" (5.7 cm) ID x 3" (7.6 cm) OD x 7" (17.8 cm) long and a hot top section 2-1/2" (6.35 cm) ID x 3" (7.6 cm) OD x 4-1/2" (11.4 cm) long.

The mold setup for the 100 pound (45.36 kg) ingots has been described previously.<sup>(1)</sup> The 15 pound (6.8 kg) mold setup was similar to the larger one.

Melt Stock - The raw materials used in melting the chromium-7% molybdenum-2% tantalum-0.09% carbon-0.10% yttrium plus lanthanum alloy are as follows:

- Chromium - "Elchrome H.P." electrolytic chromium obtained from Union Carbide Corporation
- Yttrium - Nuclear grade sponge obtained from Lunex Company
- Lanthanum - Nuclear grade ingot obtained from Lunex Company
- Molybdenum - Pressed and sintered powder obtained from General Electric Company, Refractory Metals Plant
- Tantalum - Double vacuum arc melted chips obtained from National Research Corporation
- Carbon - Spectroscopic graphite rod obtained from National Carbon.

Chemical analysis specifications and actual lot analyses of the two lots of chromium used are shown in Table I. This table also relates the lot analysis to the specific ingots made from each lot.

Vendors' lot analyses of the tantalum, molybdenum, yttrium, lanthanum, and carbon are given in Tables II through VI.

Preparation of the Charge - Because of the poor packing factor of electrolytic chromium and the resulting poor coupling obtained in induction melting, it is necessary to briquet this material. For optimum charging of the 15 pound (6.8 kg) heats, 3-3/4" (9.5 cm) diameter briquets were required. For the 100 pound (45.36 kg) heats, briquets of 2" (5.1 cm), 3" (7.6 cm), and 3-3/4" (9.5 cm) diameter were required in addition to several pounds of loose, unbriquetted material.

Because the anticipated melting problems were concerned mainly with the recovery of the carbon, yttrium, and lanthanum, the differences in melting techniques involved differences in the method of adding these elements to the molten bath.

The three variations in melting technique used required the use of three methods of charge preparation:

- a. Molybdenum, tantalum, and 0.5% yttrium were pressed into the chromium briquets and included in the initial crucible charge. This quantity of yttrium was added as a "getter" for oxygen and nitrogen and was not expected to be retained. Carbon, lanthanum, and the yttrium actually required to meet chemical specifications were added simultaneously late in the melting cycle, but as individual elements.



TABLE I. - CHEMISTRY OF "ELCHROME HP" CHROMIUM

VENDOR'S ANALYSIS - (Union Carbide Corporation)

Element	Specification Limit - wt. %	Lot 37600	Lot 37610
C	0.008 max.	0.003	0.003
S	0.008 max.	0.007	0.009
Si	0.02 max.	0.006	0.007
Fe	0.02 max.	0.009	0.012
O	0.015 max.	0.004	0.004
N	0.010 max.	0.010	0.006
H	0.0005 max.	0.001	0.001
Cr	99.9 min.	99.95+	99.95+
Lot 37600		Lot 37610	
Heat No. 983-30 Heat No. 984-30 Heat No. 994-30 Heat No. 995-30 Heat No. 996-30 Heat No. 131-100 Heat No. 138-100 Heat No. 139-100 Heat No. 140-100 (70 lb.)		Heat No. 140-100 (20 lb.) Heat No. 141-100	

TABLE II. - TANTALUM CHIPS (Ingot 7433)

VENDOR'S CHEMICAL ANALYSIS (National Research Corporation)

Element	ppm	Element	ppm
O	37	Mn	< 1
H	56	Mo	<10 (ND)
C	15	Na	--
N	18	Nb	<25
Al	<10 (ND)	Ni	9
Cr	< 1	Si	15
Cu	< 1	Sn	< 1
Fe	10	Ti	< 5
Mg	< 1	W	<40

TABLE III. - MOLYBDENUM

VENDOR'S CHEMICAL ANALYSIS (General Electric Company)

Impurity	Wt. %	
	Lot Mos34 Mix 3	Lot Mos22 Mix 2
Al	<0.001	<0.001
Ca	<0.001	<0.001
Si	0.001	0.001
Cr	<0.001	<0.001
Fe	0.001	0.002
Ni	<0.001	<0.001
Cu	<0.001	0.001
W	0.004	0.003
Mn	<0.001	<0.001
Mg	<0.001	<0.001
Sn	<0.001	<0.001
Typical Analysis (ppm)		
O	22	
N	11	
H	2	
C	15	

TABLE IV. - NUCLEAR GRADE YTTRIUM SPONGE (GE-4-67)

VENDOR'S CHEMICAL ANALYSIS (Lunex Company)

Element	ppm	Element	ppm
Aluminum	1*	Lutetium	5*
Beryllium	10*	Magnesium	---
Boron	50*	Manganese	1*
Cadmium	5*	Neodymium	---
Calcium	200**	Nickel	1*
Cerium	50*	Niobium	50*
Chromium	1*	Potassium	1*
Cobalt	1*	Praseodymium	50*
Copper	1*	Samarium	5*
Dysprosium	5*	Silicon	50*
Erbium	100*	Sodium	5*
Europium	5*	Tantalum	<50
Gadolinium	5*	Terbium	50*
Holmium	50*	Thulium	5*
Iron	---	Titanium	---
Lanthanum	---	Vanadium	1*
Lead	10*	Ytterbium	50*
Lithium	1*	Zinc	50*
		Zirconium	20*

\* No persistent line

\*\* Ca reported as 200 ppm because of electrode compartment

--- Element interfered with, no value reported.

Leco O<sub>2</sub> 480 ppmKjeldahl N<sub>2</sub> 2 ppm

TABLE V. - NUCLEAR GRADE LANTHANUM INGOT (GE-4-67)

VENDOR'S CHEMICAL ANALYSIS (Lunex Company)

Element	ppm	Element	ppm
Aluminum	1*	Lutetium	5*
Beryllium	10*	Magnesium	<30
Boron	50*	Manganese	1*
Cadmium	5*	Neodymium	50*
Cerium	50*	Nickel	1*
Calcium	200**	Niobium	50*
Chromium	1*	Potassium	1*
Cobalt	1*	Praseodymium	50*
Copper	1*	Samarium	5*
Dysprosium	5*	Silicon	50*
Erbium	100*	Sodium	5*
Europium	5*	Tantalum	10*
Gadolinium	< 15	Terbium	50*
Holmium	< 70	Thulium	5*
Iron	<100	Titanium	1*
Lanthanum	balance	Vanadium	1*
Lead	10*	Ytterbium	50*
Lithium	1*	Zinc	50*
		Zirconium	20*

\* No persistent line

\*\* Ca reported as 200 ppm because of electrode compartment

--- Element interfered with, no value reported.

Leco O<sub>2</sub> 760 ppmKjeldahl N<sub>2</sub> 4 ppm

TABLE VI. - SPECTROSCOPIC GRAPHITE AGKSP (Lot P-64)

VENDOR'S CHEMICAL ANALYSIS (NATIONAL CARBON)

Impurity	ppm Max. Concentration
Al	0.1
B	---
Ca	---
Cu	---
Fe	---
Pb	---
Mg	0.2
Mn	---
K	---
Si	0.3
Ag	---
Na	---
Sn	---
Ti	---
V	---
Total ash content	<1

All elements above were specifically sought. No other impurity lines were found and only those elements found are reported.

2. Molybdenum and 0.5% yttrium were pressed into the chromium briquets and included in the initial crucible charge. Tantalum, carbon, lanthanum, and the yttrium actually required to meet chemical specification were added simultaneously, late in the melting cycle, but as individual elements.
3. This technique was the same as Number 2 except that the late addition of tantalum, carbon, lanthanum, and yttrium was pressed into a briquet and added in this form.

The last of the three techniques provided the advantage of getting the low density and more volatile additions below the surface of the melt quickly by means of briquetting them with the high density tantalum. This was the technique used for all five 100 pound (45.36 kg) heats.

Melting Procedure - When the vacuum chamber reached a pressure of approximately 15 microns ( $2 \text{ N/m}^2$ ) of mercury in the case of the small heats and 30 microns ( $4 \text{ N/m}^2$ ) in the case of the large heats, power was applied to the hot top heater, and the entire mold setup was allowed to heat up and outgas in vacuum. When outgassing of the mold was complete, power was applied to the charge, and it was allowed to outgas in vacuum at a temperature approaching the melting point. The pressure typically increased to a value in the range of 50 microns to 100 microns ( $6.7 \text{ N/m}^2$  to  $13.4 \text{ N/m}^2$ ) and then started to decrease. Power was then shut off and the chamber was pumped to the lowest pressure attainable. Argon was then admitted to the chamber to a pressure of two-thirds of atmospheric and the charge was melted at this pressure. The charge was held molten for a period of 15 minutes to allow uniform distribution of the molybdenum and to permit the yttrium to accomplish its gettering function. At this point, the tantalum, carbon, lanthanum, and final addition of yttrium were added and the heat held molten for five minutes to insure complete homogeneity. It was then poured and cooled overnight in the vacuum chamber.

Ingot Quality - Surface condition of the ingots was very good. Figure 1 shows a typical 100 pound (45.36 kg) ingot after sandblasting.

The ingots were examined radiographically for internal defects. From 7-5/8" to 8-3/8" (19.4 cm to 21.3 cm) of shrink-free ingot was obtained in the 15 pound (6.8 kg) ingots and about 13" (33 cm) of shrink-free ingot was obtained in the 100 pound (45.36 kg) ingots. Some small gas-type porosity, not evident in the radiographs, was found just below the shrinkage cavity in the 100 pound (45.36 kg) ingots. It was located in an annular pattern at approximately the mid-radius of the ingot. It was necessary to remove approximately an additional inch (2.54 cm) from the top of the billets to eliminate this defect. Figure 2 illustrates this defect in a transverse slice taken from the top of a 100 pound (45.36 kg) ingot.



$Cr-7Mo-2Ta-.10(Y+La)-.09C$

Figure 1 Typical 100 Pound Ingot





Figure 2 Section of 100 Pound Ingot



A slice approximately 3/8" (0.95 cm) thick was cut from the top and bottom of each ingot after removal of the piped portion of the hot top. These samples were sent for chemical analysis. Results of ingot analyses are shown in Table VII.

As can be seen from this table, there is considerable variation in chemistry from heat to heat. There is no problem with tantalum, molybdenum, carbon, sulfur, or phosphorus. The inconsistent and predominantly high oxygen and zirconium contents and the equally inconsistent and low recovery of yttrium and lanthanum in the 15 pound (6.8 kg) ingots point to metal-crucible reaction as the probable source of difficulty. The 100 pound (45.36 kg) ingots have consistently lower oxygen contents and higher, but not consistent, recovery of yttrium and lanthanum. Zirconium contamination is also variable in the 100 pound (45.36 kg) heats.

It is a well-established fact that preformed crucibles exhibit great variability in properties from crucible to crucible. One of these variables is the rate of attack on the crucible by molten metal and the resulting variation from crucible to crucible in oxygen pickup in the melt. In the case of a zirconia crucible, reduction of the zirconia results in both high oxygen and zirconium in the melt.

In vacuum induction melting of more conventional materials, it has been found helpful to make a wash heat in a crucible before using. This tends to smooth out the variability from crucible to crucible. However, considering the reactivity and relatively high melting temperature of this alloy, and the condition of the crucible after making one heat, it was not considered desirable to make more than one heat per crucible.

TABLE VII. - COMPOSITION OF INGOTS AND PRODUCTS

Element	Nominal composition	Heat No. 983-30		Heat No. 984-30		Heat No. 994-30	
		Added to charge	Analyzed ingot	Added to charge	Analyzed ingot	Added to charge	Analyzed ingot
			Top Bottom		Top Bottom		Top Bottom
QUANTITATIVE ANALYSIS IN WEIGHT PERCENT							
Cr	balance	7.05	6.74 6.66	7.05	7.0 6.8	7.07	7.01
Mo	6.8-7.4	2.05	2.05 2.09	2.05	2.08 2.05	2.09	2.10
Ta	1.9-2.2	0.09	0.116 0.116	0.09	0.117 0.114	0.096	0.097
C	0.08-0.10	0.55	0.07 0.08	0.55	0.05 0.08	0.003	0.0035
Y	Total	0.20	<0.01 <0.01	0.20	<0.01 <0.01	<0.002	<0.002
L	0.08-0.12						
QUANTITATIVE ANALYSIS IN PARTS PER MILLION							
O	150 ppm max.		99 115		145 166	271	239
N	100 ppm max.		49 45		57 56	54	36
H	20 ppm max.		3 2		1 1	3	1
P	50 ppm max.		< 50 < 50		< 50 < 50	61	44
S	100 ppm max.		18 15		9 13	46	41
SPECTROGRAPHIC ANALYSIS IN PARTS PER MILLION							
Al			30 30		30 30	30	30
Fe			30 30		30 30	30	30
Hf			<500 <500		<500 <500	<500	<500
Cb			<300 <300		<300 <300	<300	<300
Ni			< 10 < 10		< 10 < 10	< 10	< 10
Si			70 70		70 70	200	200
Ti			< 10 < 10		< 10 < 10	< 10	< 10
V			< 10 < 10		< 10 < 10	< 10	< 10
W			<500 <500		<500 <500	<500	<500
Zr			70 100		< 10 < 10	100	150

TABLE VII. - COMPOSITION OF INGOTS AND PRODUCTS  
(continued)

Element	Heat No. 995-30			Heat No. 996-30			Heat No. 131-100		
	Added to charge	Analyzed ingot		Added to charge	Analyzed ingot		Added to charge	Analyzed ingot	
		Top	Bottom		Top	Bottom		Top	Bottom
QUANTITATIVE ANALYSIS IN WEIGHT PERCENT									
Cr	7.2 2.05 0.08 0.57 0.25	6.87	7.01	7.2	7.08	7.0	6.97	7.05	0.099
Mo		2.03	2.06	2.05	2.07	2.1	2.07	2.13	
Ta		0.095	0.095	0.08	0.094	0.096	0.098	0.091	
C		0.031	0.049	0.57	0.036	0.06	0.07	0.11	
Y		0.008	0.017	0.25	0.013	0.025	0.04	0.04	
L									
QUANTITATIVE ANALYSIS IN PARTS PER MILLION									
O		278	187		236	180	49	64	49 ±5
N		36	27		21	25	59	62	32 ±5
H		1	1		1	1	5	6	16 ±5
P		54	39		53	38	< 20	< 20	<20
S		90	48		23	10	26	23	<10
SPECTROGRAPHIC ANALYSIS IN PARTS PER MILLION									
Al		30	30		30	30	70	70	
Fe		30	30		30	30	30	30	
Hf		<500	<500		<500	<500	<500	<500	
Cb		<300	<300		<300	<300	<300	<300	
Ni		< 10	< 10		< 10	< 10	< 10	< 10	
Si		200	100		400	150	200	200	
Ti		< 10	< 10		< 10	< 10	10	10	
V		< 10	< 10		< 10	< 10	< 10	< 10	
W		<500	<500		<500	<500	<500	<500	
Zr		150	400		150	300	50	50	

TABLE VII. - COMPOSITION OF INGOTS AND PRODUCTS  
(continued)

Element	Heat No. 138-100			Heat No. 139-100			Heat No. 140-100		
	Added to charge	Analyzed ingot		Added to charge	Analyzed ingot		Added to charge	Analyzed ingot	
		Top	Bottom		Top	Bottom		Top	Bottom
QUANTITATIVE ANALYSIS IN WEIGHT PERCENT									
Cr	7.2	6.80	6.90	7.2	6.80	6.93	7.2	7.09	7.15
Mo	2.05	2.37	2.21	2.05	2.11	2.20	2.05	2.11	2.16
Ta	0.08	0.086	0.093	0.08	0.093	0.086	0.08	0.089	0.103
C	0.59	0.16	0.10	0.59	0.09	0.10	0.59	0.14	0.21
Y	0.25	0.075	0.065	0.25	0.11	0.11	0.25	0.12	0.12
L									
QUANTITATIVE ANALYSIS IN PARTS PER MILLION									
O		77	131		84	69		128	136
N		20	59		72	61		95	87
H		9	9		6	9		14	32
P		< 20	< 20		< 20	< 20		< 20	< 20
S		23	18		17	19		27	16
SPECTROGRAPHIC ANALYSIS IN PARTS PER MILLION									
Al		20	20		20	20		20	20
Fe		30	30		30	30		30	30
Hf		< 500	< 500		< 500	< 500		< 500	< 500
Cb		< 300	< 300		< 300	< 300		< 300	< 300
Ni		< 10	< 10		< 10	< 10		< 10	< 10
Si		100	100		100	100		100	100
Ti		30	30		< 10	< 10		< 10	< 10
V		< 10	< 10		< 10	< 10		< 10	< 10
W		< 500	< 500		< 500	< 500		< 500	< 500
Zr		200	200		30	30		70	70

(1) Replicate analysis

TABLE VII. - COMPOSITION OF INGOTS AND PRODUCTS  
(continued)

Element	Heat No. 141-100			Product
	Added to charge	Analyzed ingot		
		Top	Bottom	
Cr	7.2	7.15	7.02	0.123
Mo	2.05	2.00	2.15	
Ta	0.08	0.075	0.112	
C	0.59	0.11	0.14	
Y	0.25	0.10	0.12	
L				
O		35	42	30, 24(1) 41, 47(1) 24, 26(1) < 20 < 10
N		61	59	
H		5	6	
P		< 20	< 20	
S		10	45	
Al		50	50	
Fe		70	70	
Hf		<500	<500	
Cb		<300	<300	
Ni		< 10	< 10	
Si		100	100	
Ti		< 10	< 10	
V		< 10	< 10	
W		<500	<500	
Zr		50	50	



## EXTRUSION

Preparation of Ingots for Extrusion - Following radiography, the hot top was removed from each ingot by cutting on a power hack saw at a point just below the bottom of the primary shrink cavity. Transverse slices for chemical analysis were then sawed as described under Ingot Quality. All sawcut faces were examined by the "Dye-chek" technique, to reveal any small cracks which would probably not be detected by radiography, or which could be generated by the sawing operation. On the 100 pound (45.36 kg) ingots, additional cropping of 3/4" to 1" (1.90 cm to 2.54 cm) of material was done on the top ends of the ingots to remove the annular porosity (see Figure 2), and an additional loss of 1/2" to 3/4" (1.27 cm to 1.90 cm) of length was incurred at the bottom ends in removing material containing hairline cracks, presumably formed during cooling of the ingot in the mold.

After cropping was completed, the 15 pound (6.8 kg) heats ranged from 6-1/4" to 7" (15.9 cm to 17.8 cm) in length, and weighed 6-1/2 pounds to 7-1/2 pounds (3.94 kg to 3.4 kg). The 100 pound (45.36 kg) heats, after cropping, ranged from 10.65" to 11.7" (27.1 cm to 29.7 cm) in length, and weighed between 55 pounds and 61 pounds (24.9 kg and 27.6 kg). These ingots were lathe-turned to remove the relatively rough cast surface and to provide the proper size match with a molybdenum extrusion can. Each 15 pound (6.8 kg) heat was turned to 1.937" (4.9 cm) and cut into two extrusion billets. The large heats were turned to 4.700" to 4.800" (11.9 cm to 12.2 cm) diameter, varying slightly with the surface condition. A 45° (0.786 radian) chamfer was machined on one end of each billet; the chamfered end then became the nose or leading end of the extrusion. For all extrusions, the nose of the extrusion was the end of the billet which was nearer the bottom of the ingot. With very few exceptions, the machined ingot surfaces were very good. After lathe-turning, a few of the large ingots showed a small number of pinhole-size defects, which were carefully removed with a hand grinder.

At the completion of conditioning, billets from the small ingots (two billets per ingot) ranged from 2.9" to 3.45" (7.4 cm to 8.8 cm) in length and weighed from 2.3 pounds to 2.7 pounds (1.04 kg to 1.22 kg). Billets from the larger heats ranged from 10.65" to 11.7" (27.1 cm to 29.8 cm) in length, and weighed from 50.2 pounds to 58.4 pounds (22.75 kg to 26.45 kg).

Canning - Extrusion cans for all billets were made from molybdenum powder by hydrostatic pressing, sintering, and finally machining to fit each billet. Just prior to extrusion, the billet, the molybdenum can, and two molybdenum tail plugs were thoroughly degreased, assembled, and loaded into an inert atmosphere welding chamber. The chamber was pumped down to a pressure of approximately 20 microns (2.67 N/m<sup>2</sup>). Immediately before welding, the chamber was back-filled with helium to a pressure of one atmosphere (1.01 x 10<sup>5</sup> N/m<sup>2</sup>), pumped down to approximately 20 microns (2.67 N/m<sup>2</sup>), and again back-filled to one atmosphere (1.01 x 10<sup>5</sup> N/m<sup>2</sup>) with helium. The molybdenum can was then sealed using a tungsten electrode by making a circumferential arc weld between the can and one of the molybdenum plugs which had been inserted into the can at the rear end of the extrusion billet. The welded assembly, ready for extrusion,

is shown in Figure 3 for a nominal 5" (12.7 cm) diameter billet. The configuration for a nominal 2" (5.08 cm) diameter billet is very similar.

The smaller billets, canned and ready for extrusion, varied from 3.83" to 4.37" (9.75 cm to 11.1 cm) in length and weighed from 3.73 pounds to 4.2 pounds (1.69 kg to 1.90 kg). The larger billets ranged from 13" to 14.06" (33.0 cm to 35.7 cm) in length and weighed from 74.7 pounds to 79.5 pounds (33.9 kg to 36.0 kg).

Extrusion - The canned billets were extruded through hot work (H-21) steel dies having a conical entry and rectangular orifice. The dies were coated by plasma spraying with zirconia to a nominal thickness of 0.015" (0.38 mm). Fiske 604 and either Corning 7052 or 7740 glass were used as lubricants. Billets were heated to the extrusion temperature in a resistance-type furnace with a H<sub>2</sub> atmosphere, transferred to a horizontal, 1250-ton Loewy press, and extruded. The resulting data are given in Table VIII. Transfer times (elapsed time from billet leaving furnace to application of extrusion force) ranged from 11 seconds to 14 seconds for the smaller billets, and from 23 seconds to 27 seconds for the larger diameter billets. Experience with a chromium-5% tungsten-.07% yttrium alloy<sup>(1)</sup> had indicated the desirability of using a post-extrusion stress relief. Since the chromium-7% molybdenum-2% tantalum alloy was expected to be stronger and probably less ductile than the 5% tungsten alloy, all extruded bars were taken from the press runout table while still hot, placed in a furnace at 1920°F (1320°K), held for approximately one hour at 1920°F (1320°K), then furnace cooled to 480°F to 570°F (522°K to 573°K) in 40 to 44 hours, removed from the furnace, and sand cooled to room temperature.

Despite the use of this stress relief, some hairline cracks appeared at the nose end of each extruded bar. The relatively large amount of molybdenum present at the nose, coupled with the fact that chromium has a larger coefficient of thermal expansion than molybdenum, resulted in the presence, upon cooling, of a residual tensile stress in the chromium alloy at the nose, causing the cracking. The geometric pattern of the cracks was the same in both large and small bars. Fortunately, these cracks did not propagate beyond the point at which the chromium alloy core assumed its full dimensions inside the jacket; therefore, this cracking resulted in the loss of no more material than that which was routinely cropped from the noses of the extruded bars.

An excellent bond was obtained between the chromium alloy and the molybdenum cladding on all sound extruded material. The molybdenum was left on the bars throughout the subsequent processing.

Ten 2-1/16" (5.24 cm) diameter billets were extruded as part of Task I, a limited exploration of some of the primary extrusion variables. The extrusion ratios chosen, nominally 3.7 and 4.7, represented a compromise among the expected deformation characteristics of the alloy, the desire to have, as a minimum, a ratio in the vicinity of four, and the size requirements for the material to be produced in Task II. In addition, the requirements imposed by the Task II rolling process parameters had to be considered. For example, the



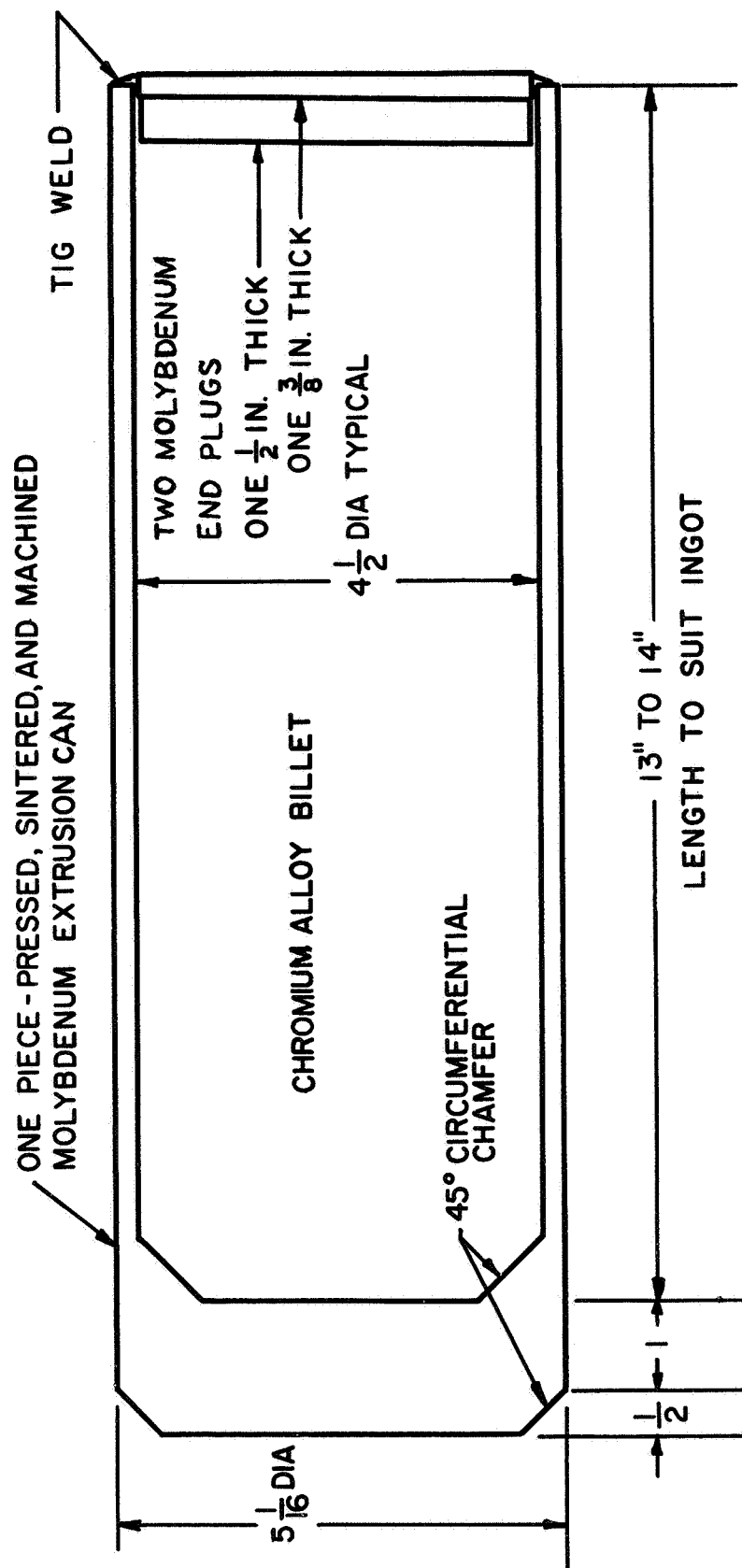


FIGURE 3 SCHEMATIC DIAGRAM OF TYPICAL EXTRUSION CAN AND BILLET ASSEMBLY

TABLE VIII. - EXTRUSION DATA

Billet number	Billet diameter	Extrusion ratio	Extrusion temperature	Breakthrough force	"K" factor
983-30T	2-1/16" (5.24 cm.)	4.9	2818°F (1820°K)	222 tons (1.975x10 <sup>6</sup> N)	79,300psi (5.47x10 <sup>8</sup> N/m <sup>2</sup> )
983-30B	2-1/16" (5.24 cm.)	3.9	2818°F (1820°K)	185 tons (1.645x10 <sup>6</sup> N)	77,200psi (5.33x10 <sup>8</sup> N/m <sup>2</sup> )
984-30T	2-1/16" (5.24 cm.)	3.65	2678°F (1740°K)	203 tons (1.805x10 <sup>6</sup> N)	89,000psi (6.14x10 <sup>8</sup> N/m <sup>2</sup> )
984-30B	2-1/16" (5.24 cm.)	4.4	2678°F (1740°K)	231 tons (2.055x10 <sup>6</sup> N)	88,700psi (6.11x10 <sup>8</sup> N/m <sup>2</sup> )
994-30T	2-1/16" (5.24 cm.)	3.5	2910°F (1870°K)	218 tons (1.940x10 <sup>6</sup> N)	98,900psi (6.82x10 <sup>8</sup> N/m <sup>2</sup> )
994-30B	2-1/16" (5.24 cm.)	3.55	2824°F (1825°K)	185 tons (1.645x10 <sup>6</sup> N)	83,000psi (5.71x10 <sup>8</sup> N/m <sup>2</sup> )
995-30T	2-1/16" (5.24 cm.)	4.4	2824°F (1825°K)	222 tons (1.975x10 <sup>6</sup> N)	85,200psi (5.87x10 <sup>8</sup> N/m <sup>2</sup> )
995-30B	2-1/16" (5.24 cm.)	4.24	2863°F (1845°K)	234 tons (2.080x10 <sup>6</sup> N)	92,000psi (6.34x10 <sup>8</sup> N/m <sup>2</sup> )
996-30T	2-1/16" (5.24 cm.)	3.6	2698°F (1755°K)	215 tons (1.910x10 <sup>6</sup> N)	95,400psi (6.58x10 <sup>8</sup> N/m <sup>2</sup> )
996-30B	2-1/16" (5.24 cm.)	4.4	2698°F (1755°K)	246 tons (2.190x10 <sup>6</sup> N)	94,300psi (6.51x10 <sup>8</sup> N/m <sup>2</sup> )
131-100	5-1/16" (12.85 cm.)	4.45	2790°F (1805°K)	1059 tons (9.43x10 <sup>6</sup> N)	67,000psi (4.62x10 <sup>8</sup> N/m <sup>2</sup> )
138-100	5-1/16" (12.85 cm.)	4.39	2696°F (1755°K)	1131 tons (1.01x10 <sup>7</sup> N)	72,300psi (4.99x10 <sup>8</sup> N/m <sup>2</sup> )
139-100	5-1/16" (12.85 cm.)	4.42	2620°F (1710°K)	1180 tons (1.05x10 <sup>7</sup> N)	75,200psi (5.18x10 <sup>8</sup> N/m <sup>2</sup> )
140-100	5-1/16" (12.85 cm.)	4.39	2687°F (1750°K)	1101 tons (9.81x10 <sup>6</sup> N)	70,400psi (4.86x10 <sup>8</sup> N/m <sup>2</sup> )
141-100	5-1/16" (12.85 cm.)	4.43	2678°F (1740°K)	1068 tons (9.51x10 <sup>6</sup> N)	67,900psi (4.68x10 <sup>8</sup> N/m <sup>2</sup> )

expected desirability of imparting large amounts of reduction (as high as 90%) in the rolling process, required that the extrusions ultimately produced from the larger diameter Task II billets be a minimum of 1-1/8" (2.86 cm) thick (including molybdenum cladding thickness). One additional factor was that any appropriate combination of extrusion variables determined in the 2-1/8" (5.4 cm) diameter extrusion container, where a pressure in excess of 180,000 psi ( $1.24 \times 10^9$  N/m<sup>2</sup>) can be generated on the billet, would have to be translatable to the 5-3/16" (13.15 cm) container, in which the maximum pressure which can be generated on a billet is approximately 120,000 psi ( $8.3 \times 10^8$  N/m<sup>2</sup>).

Six combinations of extrusion ratio and billet temperature were employed on the ten 2-1/16" (5.24 cm) diameter billets. Examination of the resulting microstructures at Lamp Metals and Components Department, Cleveland, Ohio, failed to reveal significant differences between these extrusions. The only problem encountered was in conjunction with Billet 994-30T, which was heated to a higher temperature, 2910°F (1870°K), than the other billets. This extrusion exhibited numerous large, transverse failures extending well in toward the center of the bar. It appeared as though the failures were intergranular in nature and that the bar had literally torn open at the grain boundaries. Thus, under the extrusion conditions employed for the smaller billets, 2863°F (1845°K) was the highest temperature successfully attempted. "K" values obtained in Task I indicated that, based on the lower unit pressure available in the larger container, an extrusion ratio of approximately five would be the maximum attainable with the larger billets. It was realized that the 5-1/16" (12.85 cm) diameter billets would have lower "K" factors than those obtained with the smaller billets, because of better heat retention in the larger billets, and because the lower surface area/volume ratio of the larger billet results in a decrease in that portion of the extrusion force required to overcome friction. Therefore, it would have been reasonable to anticipate a slightly higher ratio than the predicted maximum of five. However, a higher ratio would have produced a bar with cross section smaller than the optimum for subsequent rolling. Accordingly, a die was designed based on a ratio of 4.6.

Three different billet temperatures, two ram speeds, and a pre-extrusion solution treatment were incorporated into the processing of the five larger diameter billets in an effort to obtain completely sound extrusions. However, the tearing type of defect discussed above was present, in varying degree, in each of the five bars produced.

It was recognized that, for the same set of conditions, the maximum temperature to which billet could be heated for extrusion without encountering the severe tearing previously discussed would be lower for the 5-1/16" (12.85 cm) than for the 2-1/16" (5.24 cm) billets, because of better heat retention in the larger billets. Accordingly, a nominal temperature of 2775°F (1795°K) was chosen for extrusion of the first large billet, as a compromise between possibly prohibitive container pressures at lower temperatures and severe tearing at higher temperatures. The billet was actually extruded at 2790°F (1805°K); unfortunately, this temperature did not prove to be low enough to avoid tearing on a 5-1/16" (12.85 cm) billet. The resulting bar had numerous

tears beginning approximately 12" (30.5 cm) from the nose, and increasing in frequency toward the tail of the bar, as though the heat produced by deformation were accumulating in the billet faster than it could be dissipated.

The container pressure required for this extrusion (approximately 100,000 psi or  $6.9 \times 10^8 \text{ N/m}^2$ ) was, as anticipated, somewhat lower than the pressures needed for the 2-1/16" (5.24 cm) billets. Although the figure of 100,000 psi ( $6.9 \times 10^8 \text{ N/m}^2$ ) represents approximately 83% of the available pressure for the larger billets, it did afford some latitude for lowering the extrusion temperature. It was decided to extrude the next billet at, nominally, 2687°F (1745°K). In addition, J. W. Clark, Flight Propulsion Division, General Electric Company, suggested, on the basis of his experience with this alloy, that a pre-extrusion solution treatment at approximately 2825°F (1825°K) might be beneficial to the extrusion characteristics of the alloy; so, it was decided to attempt this also. The billet was heated to 2820°F (1810°K) and held for approximately two hours. The temperature was then dropped to 2696°F (1755°K), allowed to equilibrate, and the billet was extruded. The changes discussed did not eliminate the tearing phenomenon, but did reduce it substantially, and a much greater yield of sound material was realized.

A further reduction in extrusion temperature appeared possible, and it seemed that this would eliminate the tearing. The third 5-1/16" (12.85 cm) billet was also solutioned prior to extrusion and was extruded at 2620°F (1710°K). However, instead of the tearing being eliminated, it was more severe than on the previous bar. For the fourth large billet, the solution treatment was discontinued, and since the best extrusion to date had been made at 2696°F (1755°K), that temperature was employed again. This resulted in the best of the five large extrusions, showing only a few tears near the tail and some minor tearing at spots on the corners of the bar; thus, it was decided to extrude the final billet under the same conditions, except for a decrease in ram speed. This was done to allow more time for the heat produced by deformation to be dissipated during extrusion, hopefully keeping the internal temperature of the billet down, decreasing the tendency for the material to tear. Even though this final 5-1/16" (12.85 cm) extrusion was made at a ram speed (1"/second or 2.54 cm/second), only slightly over half that of the other large billets (1.8"/second or 4.66 cm/second), it was torn severely over the rear half of the bar. This was all too typical of these defects; that is, there seemed to be no particular pattern to their frequency or location, and furthermore, little sensible correlation with extrusion parameters. The defects did not seem to be due to lubrication failures, since, except for the torn areas, the surfaces of the extruded bars were quite smooth over their entire length. Also, the random occurrence of the defects did not suggest inadequate lubrication. Neither could the defects be correlated with the post-extrusion condition of the die coating.

Perhaps, during cooling of the larger ingots in the mold, some segregation or solid state reaction occurs which results in the formation of an undesirable grain boundary constituent - one which might not have time to form in the more rapidly cooling smaller ingots - and which might have an adverse effect on the workability of the alloy.

Although the  $ZrO_2$  die coating did spall to the extent that the dies had to be sandblasted clean and recoated after each extrusion, the coatings did an excellent job of protecting the surfaces of the dies from wearing excessively or washing out, and in preventing pickup of die steel on the extrusions. Two dies were used for the ten small extrusions; two were needed only because two different extrusion ratios were desired. One die was used for all five large extrusions.

The rectangular bars extruded from the smaller billets were of two nominal sizes:

1. 0.625" thick x 1.5" wide x 13" to 15" long (1.59 cm x 3.81 cm x 33 cm to 38 cm)
2. 0.500" thick x 1.5" wide x 16" to 19" long (1.27 cm x 3.81 cm x 40.6 cm to 48.3 cm).

On a given bar, the average thickness variation was approximately 0.020" (0.51 mm) and the average width variation approximately 0.012" (0.3 mm). Bars extruded from the larger billets were nominally 1.125" thick x 4.125" wide x 55" to 59" long (2.86 cm x 10.5 cm x 140 cm to 150 cm). Average variation in thickness on any given bar was approximately 0.022" (0.56 mm); average width variation was approximately 0.037" (0.94 mm).

Discussion of Extrusion Data - Billet diameters shown include the thickness of the molybdenum jacket. The numbers for the 2-1/16" (5.24 cm) diameter billets have "T" and "B" suffixes; these designate top half and bottom half of an ingot. Extrusion temperatures were determined with an optical pyrometer. The minor variations from the intended extrusion ratios of 3.7 and 4.7 for the smaller billets and from 4.6 for the larger billets are due to differences in the thickness of the  $ZrO_2$  coating which was plasma sprayed on the die face and throat. The dies were sandblasted clean and recoated after each extrusion. It was difficult to deposit a coating of the same thickness each time, particularly in the case of the smaller dies, which, of course, had much smaller apertures.

The "K" factor, or "extrusion constant," is based on the breakthrough force and was calculated from the equation  $P = K \cdot \ln R$  where  $P$  = extrusion pressure and  $R$  extrusion ratio. Calculated "K" values for the 2-1/16" (5.24 cm) diameter billets look reasonable when plotted versus extrusion temperature (Figure 4), with the exception of Billets 995-30B and 994-30T. These two billets, although extruded at higher temperatures than the others, had higher, rather than lower, "K" factors. However, these were the only two of the ten small billets which were extruded after a new liner was installed in the extrusion container. The old liner was worn approximately 0.020" (0.51 mm); thus, the new liner resulted in a significantly-reduced clearance between the liner and the hot billet, thereby reducing the amount of lubricant which can be retained between billet and liner. This could be detrimental in two ways. First, a significant decrease in the amount of lubricant between

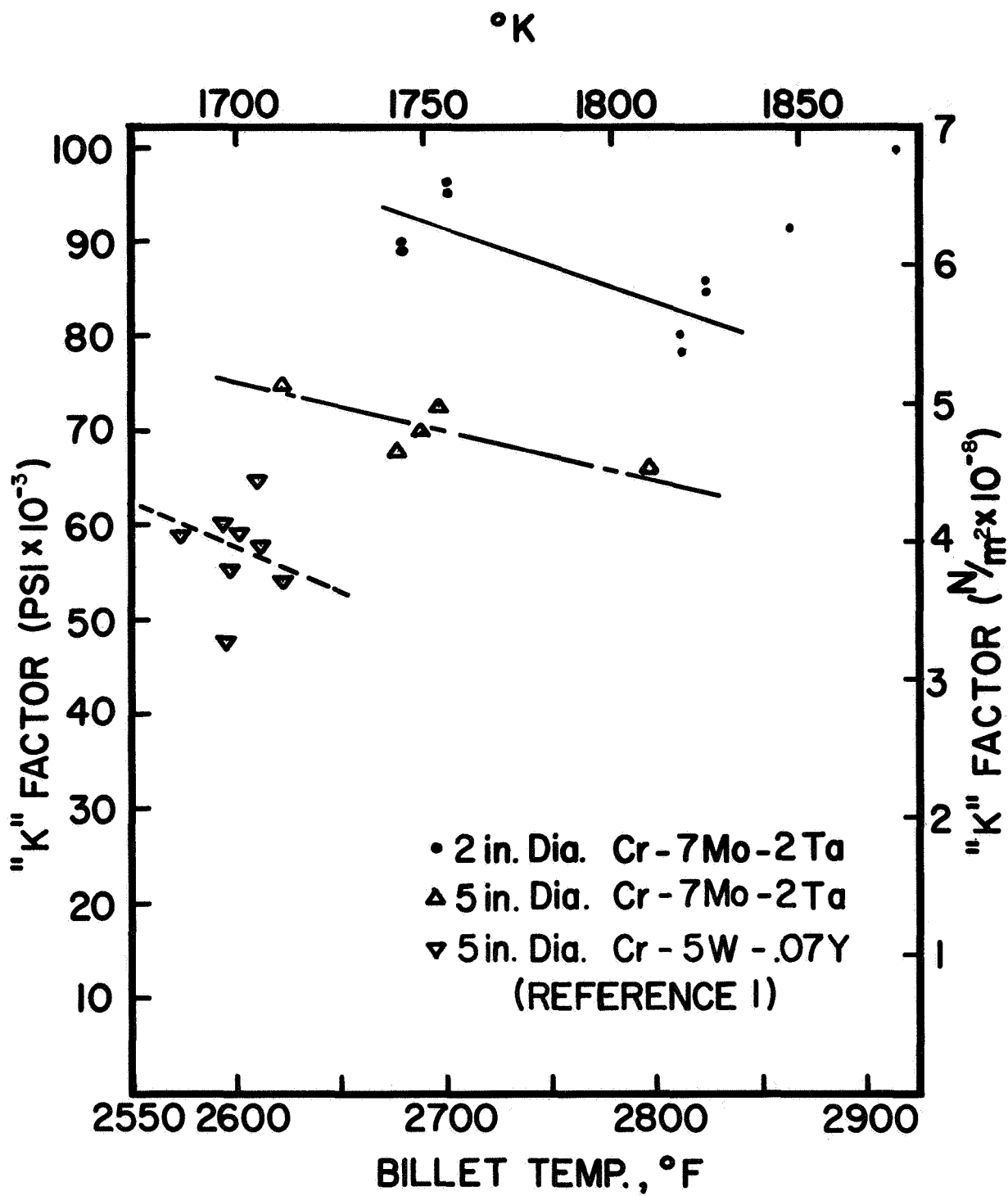


Figure 4 Extrusion Constant

the billet and liner could result in an increase in the frictional forces accompanying extrusion; second, since one of the functions of a high-temperature lubricant is to provide thermal insulation between workpiece and tooling, insertion of the new smaller diameter liner could have resulted in a higher rate of heat transfer from the hot billet to the liner. At billet temperatures on the order of 2900°F (1865°K), even though the billet rests in the liner only about four seconds before extrusion commences, the new liner probably results in a significant increase in heat loss from the billet and a corresponding significant increase in resistance to deformation; thus, an abnormally high "K" factor may be obtained.

The "K" values obtained for the larger diameter billets appeared to vary with temperature in a reasonable manner (again, Figure 4). Billet 141-100 had a "K" factor which is somewhat below the curve established by the four previous large billets, but this is due to the fact that it was extruded at a slower speed, resulting in a somewhat lower breakthrough force. For comparison with data from the larger diameter billets, Figure 4 includes data from extrusion of 5-1/16" (12.85 cm) diameter billets of chromium-5% tungsten-0.07% yttrium, carried out at this laboratory under a previous NASA contract.<sup>(1)</sup> It is apparent that the anticipated higher strength of the chromium-7% molybdenum-2% tantalum alloy is reflected in a "K" factor significantly higher [at least in the neighborhood of 2600°F (1700°K)] than that of the chromium-5% tungsten-0.07% yttrium alloy.





## ROLLING AND ANNEALING

Goals - The scope of work specified in the contract established goals for the rolling of the chromium alloy. The requirements for finish and dimensional tolerances were not especially stringent and would normally be met by ordinary care. The goal for the yield of 106 pounds (48 kg) of product was consistent with the yields obtained in a previous program at this facility for a chromium-5% tungsten alloy. Requirements for mechanical properties were not specified.

The prevention of cracking during rolling, which would be essential in meeting the requirement for yield, was quickly identified as the major problem by the results of the first few rolling experiments. A satisfactory jacket and the counteraction of the effects of the differential thermal contraction between the chromium alloy and the jacketing materials proved to be as important as the rolling and annealing practices in preventing cracking. Since most developmental effort was needed to obtain adequate yields and the scope of the work did not anticipate efforts to improve mechanical properties, any experimental work to improve mechanical properties had to be incidental. However, there was an opportunity to try to improve a specific mechanical property without going beyond the scope of the program. This occurred late in Task 1 while the first of the 100 pound (45 kg) melts was being processed through the extrusion operation.

A low ductile to brittle bend transition temperature (DBBTT) is only one of several mechanical properties of the alloy that would be important in any probable application. However, the DBBTT was the only property that could be determined because of limitations in time and effort.

Annealing of the Extrusions - The annealing of an extrusion from a 15 pound (6 kg) melt was studied to find the most easily worked starting condition. The structure as extruded was almost completely recrystallized with some large, slightly wrought grains. The annealing conditions were the factorial combinations of five temperatures and two times. The effects were evaluated metallographically and by hardness measurements.

The hardness was affected only slightly by the annealing, but there was a minimum for the one hour treatment at 2552°F (1673°K) as shown below:

Annealing temperature	Average hardness, Rockwell 30T annealing time	
	<u>One hour</u>	<u>Five hours</u>
2192°F (1473°K)	84.0	82.6
2372°F (1573°K)	82.7	83.2
2552°F (1673°K)	82.0	82.4
2732°F (1773°K)	83.5	83.2
as extruded	84.2	

Growth of precipitate particles, which were presumed to be tantalum carbide, apparently occurred within one hour at 2372°F (1573°K) and 2552°F (1673°K). There appeared to be some solution and precipitation of finer particles at the highest temperature, 2732°F (1773°K), in one hour and at 2552°F (1673°K) in five hours. All of the treatments recrystallized the structure completely, and secondary grain growth occurred during the 2732°F (1773°K) treatments.

The effects of only one treatment, one hour at 2552°F (1673°K), were evaluated further by elevated temperature tensile tests (Table IX and Figures 5 to 7). Since this treatment caused a minimum in hardness and increased the ductility of the alloy somewhat at temperatures anticipated to be in the rolling temperature range, it was adopted as the standard treatment for all extrusions.

Jacketing - Jacketing of the alloy was essential in preventing cracking, but it had harmful as well as beneficial effects. Jacketing reduced cracking by preventing the embrittlement of the alloy by the reaction with oxygen or nitrogen at elevated temperatures and by providing some mechanical restraint to the edges of the strip during rolling. Jacketing tended to cause cracking because of the differential thermal contraction between the chromium alloy and its jacket upon cooling and because of nonuniform deformation of the chromium alloy during rolling.

In a previous program at this facility, it was found that an oxide film sufficiently protective to allow several warm rolling passes to be made without cracking could be formed on the chromium-5% tungsten alloy. Therefore, an attempt was made to duplicate that process with the present alloy. The alloy was ground and electro-etched to prepare the surface for oxidation. An oxide film was formed by exposing the alloy to wet hydrogen at 2012°F (1373°K) for 15 minutes. The alloy cracked badly during the first rolling pass at 2372°F (1573°K). If the alloy is protected from contamination, it does not crack under these rolling conditions.

Since there was a reluctance to take unnecessary steps to prevent contamination by the atmosphere, the use of an evacuated jacket was started after several less costly methods proved to be inadequate. It was possible to assign contamination as the cause of certain cracks that occurred with the less effective jackets since those cracks occurred only where the chromium alloy was discolored and not where the alloy had been protected sufficiently to prevent discoloration. A jacket similar to that shown in Figure 8, but with only intermittent welds and not evacuated, was adequate protection for the Task 1 extrusions; however, it was not adequate for Task 2 extrusions, which were larger and consequently required longer times for heating. All but one of the packs for the large extrusions were evacuated.

TABLE IX. - TENSILE PROPERTIES  
OF EXTRUSIONS AND SHEET

Extrusion number	Condition	Testing temperature F	Testing temperature K	Ultimate strength psi N/mm <sup>2</sup> (a)	Yield strength 0.2% offset psi N/mm <sup>2</sup>	Elongation %	Reduction in area %
983-30B ↓	As extruded	932	773	69.2 477	61.0 420	0	1
		1292	973	66.4 457	56.3 388	1	1
		1652	1173	77.3 533	52.9 365	3	2
		1832	1273	78.9 543	49.1 338	23	60
		2012	1373	61.2 422	45.0 310	20	50
	Extruded and annealed at 2552°F (1673°K) for one hour	932	773	65.6 451	62.0 427	0	1
		1112	873	68.2 470	57.4 395	1	1
		1292	973	74.5 514	54.1 373	2	2
		1472	1073	72.0 496	54.2 374	2	2
		1652	1173	87.2 601	55.5 382	5	5
131-100 ↓	Process N (Reference process for 1/16" sheet pro- duct)	572	573	51.7 356	--	0	1
		752	673	83.7 577	48.6 345	14	10
		1292	973	80.0 551	42.4 292	20	43
		1652	1173	67.2 464	35.6 245	32	54
	Process N and annealed at 2462°F (1623°K) for one hour (1/16" sheet)	1112	873	93.4 643	52.2 360	10	5
		1472	1073	89.1 615	45.8 316	18	29
		1900	1311	71.1 490	41.7 287	16	34
		2012	1373	46.9 323	22.7 156	40	54
		2100	1422	42.1 290	27.7 191	44	63

<sup>a</sup>All tests above room temperature were conducted in vacuum. The crosshead speed of the testing machine was 0.0025" per minute ( $1.04 \times 10^{-6}$  m/s) to a strain of 0.6%, then speed was increased by a factor of ten. The gage length was 0.5" (1.27 cm).

TABLE IX. - TENSILE PROPERTIES OF EXTRUSIONS  
AND SHEET (Continued)

Extrusion number	Condition	Testing temperature F K	Ultimate strength thousands psi N/mm <sup>2</sup> (a)	Yield strength 0.2% offset psi N/mm <sup>2</sup>	Elongation %	Reduction in area %
138-100	Process N (reference process for 1/16" product) (1/16" sheet)	Room temperature	Specimen broke during loading of testing machine			
			45.2	312	24.4	168
			35.3	243	19.6	135
			19.0	131	7.6	52
					34	50
140-100	Process N (reference process for 1/16" product) (1/16" sheet)	Room temperature	Specimen broke during loading of testing machine			
			47.1	325	28.0	193
			32.6	224	19.1	131
			15.5	107	5.6	39
					40	39

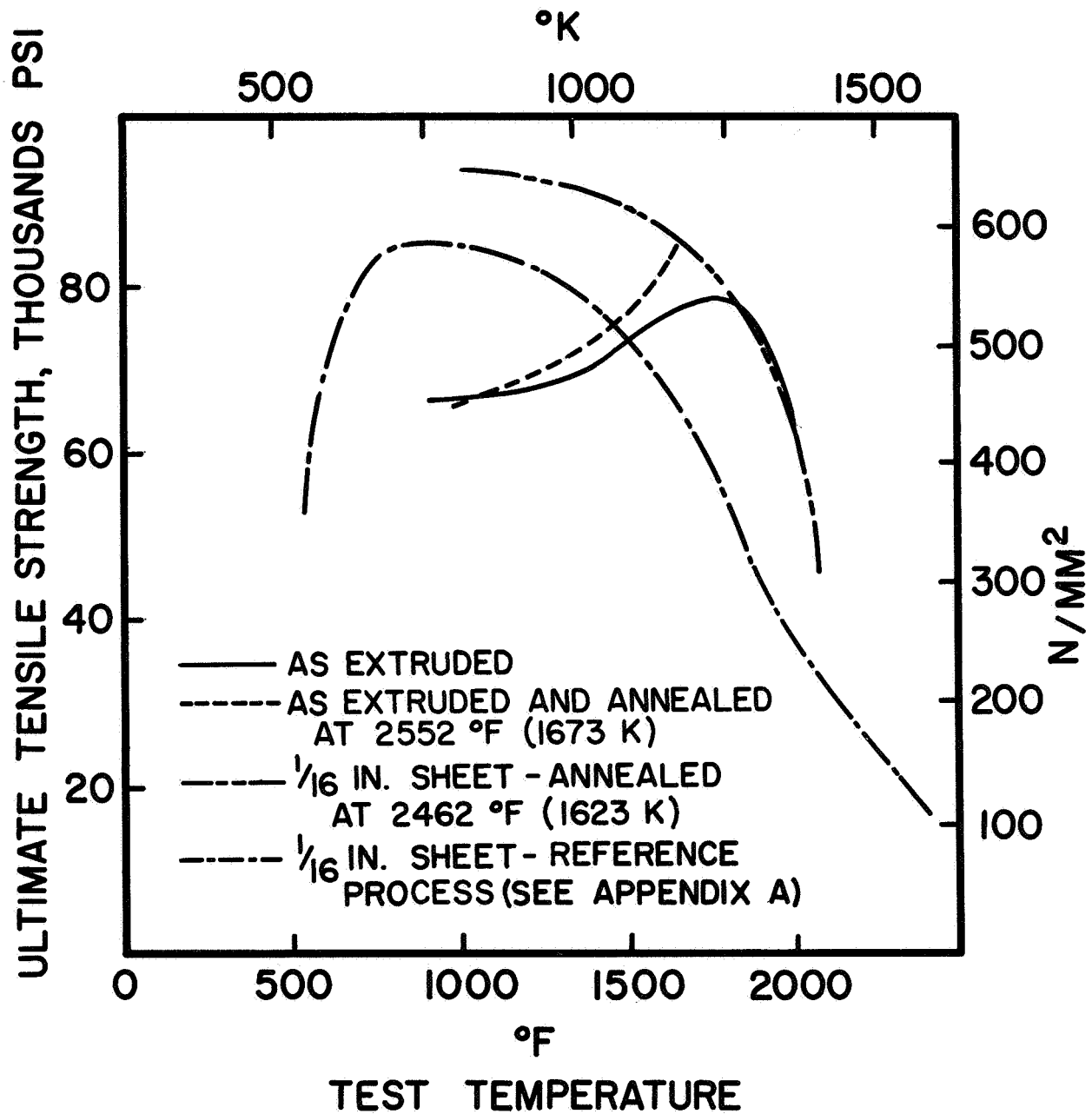


Figure 5 Ultimate Tensile Strength of Extrusions and Sheet

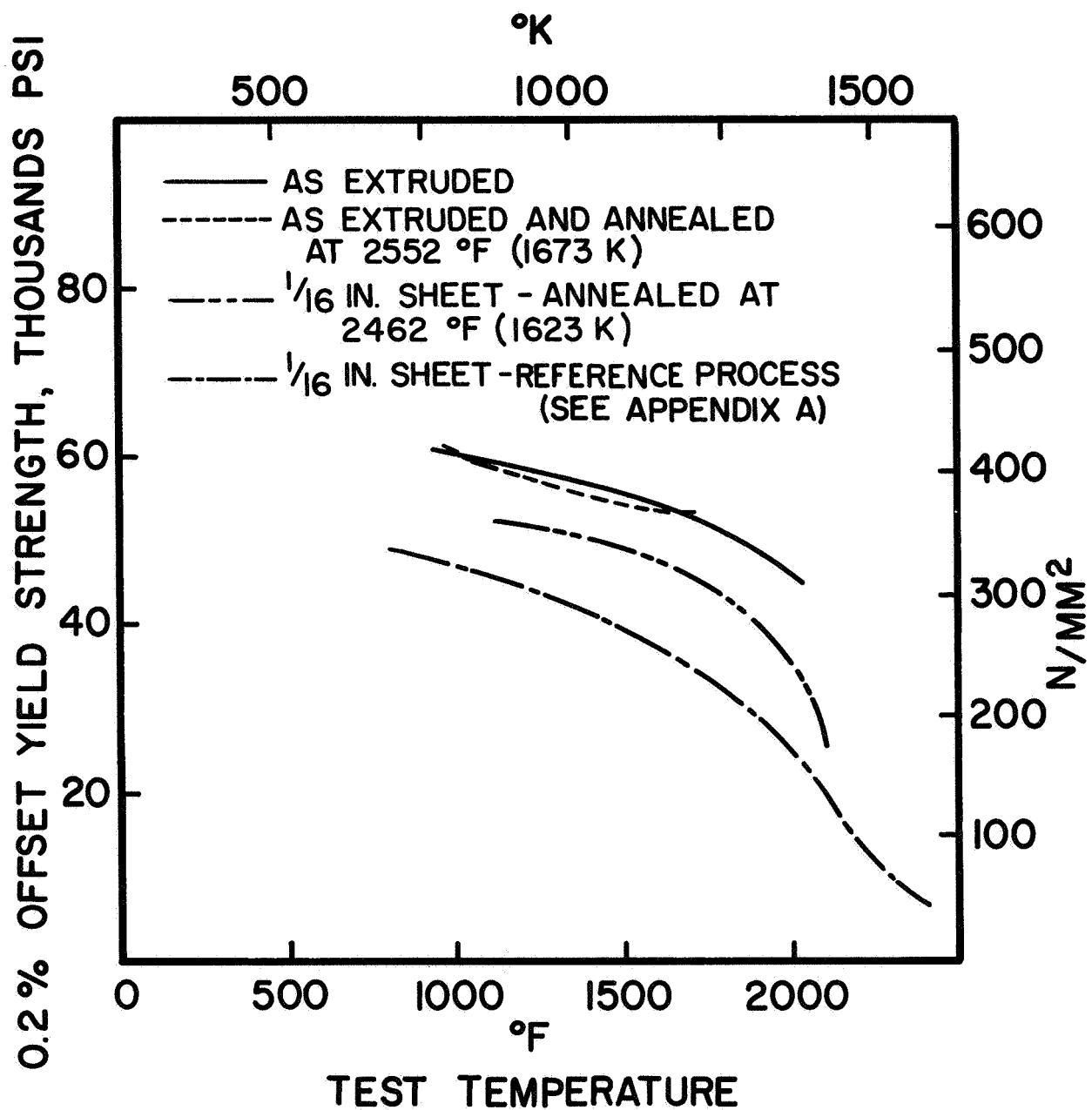


Figure 6 Yield Strength of Extrusions and Sheet

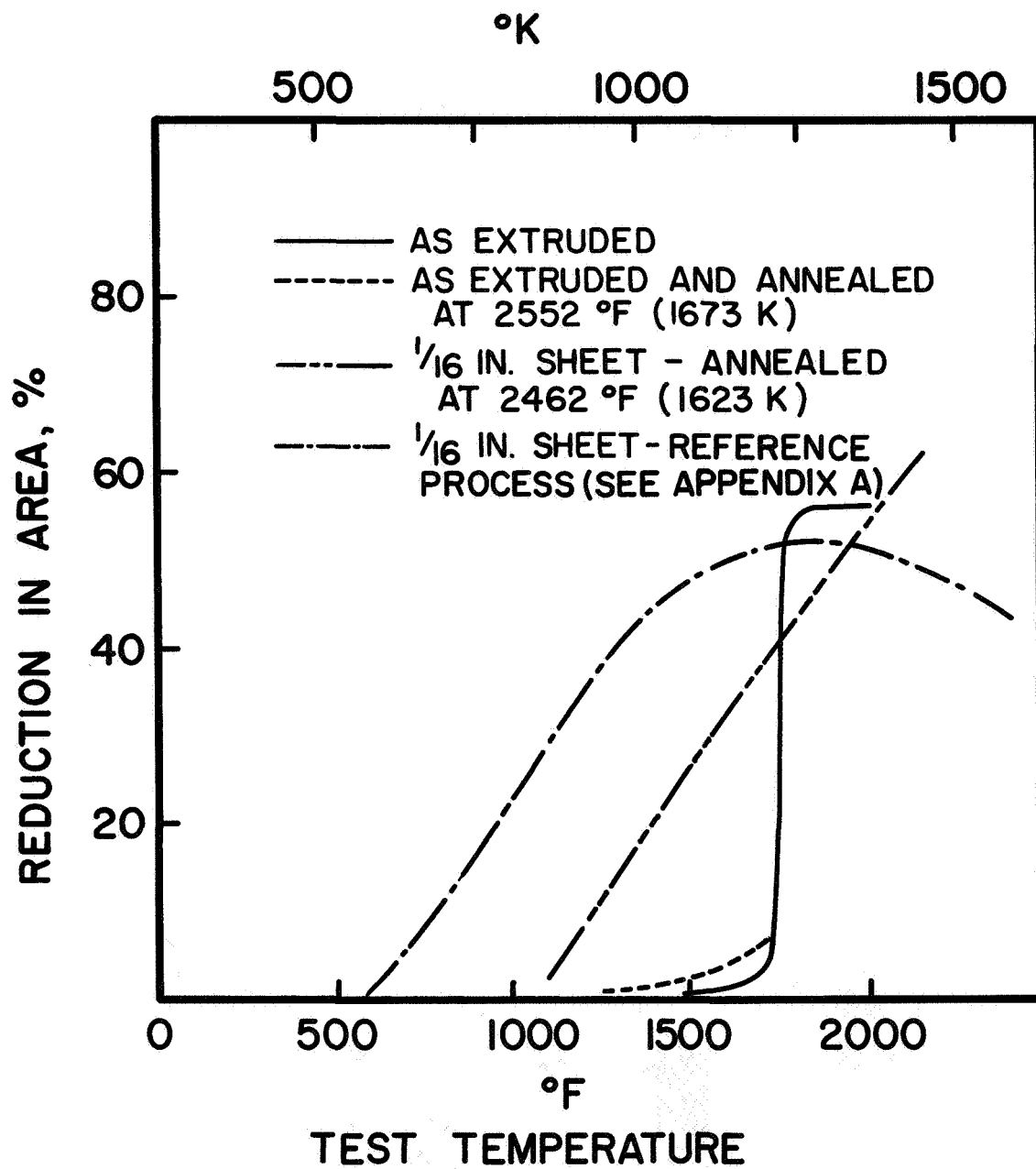


Figure 7 Ductility of Extrusions and Sheet

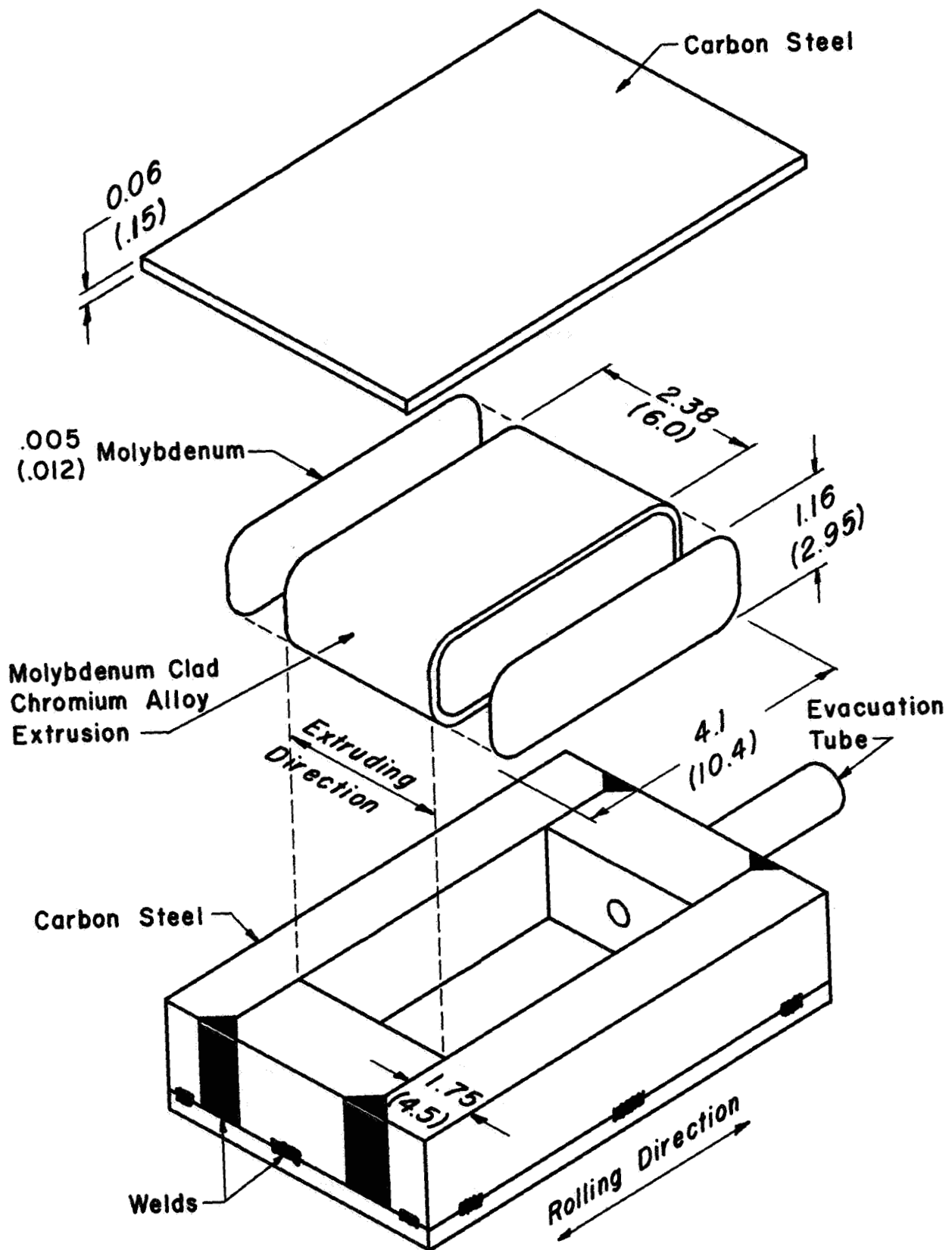


Figure 8 Exploded View of Pack



The packs, i.e., the assembly of the extrusion and its jacket, were always heated for rolling and annealing in hydrogen atmosphere furnaces and were typically exposed to air during rolling for thirty seconds or less per rolling pass. The furnace atmospheres contained water vapor principally from back streaming through flame curtains and from infiltration and nitrogen from those sources and an auxiliary nitrogen curtain at one of the furnace doors.

The mechanical restraint of the edges of the chromium alloy strip by the steel bars at the sides of the jackets apparently tended to reduce edge cracking of the chromium alloy during rolling. Since the amount of material for process development was quite limited and it was thought that the restraint offered by the side bars was less important than other factors in preventing cracking, the effect of the width of the side bars was not studied systematically. However, where the width of the side bars, as well as other factors, were varied in the early rolling experiments, there was a tendency for the packs with the widest side bars to have the least cracking of the chromium alloy. No controlled experiments were performed to confirm this relationship, but in one series of two experiments, only the final annealing conditions and width of the steel bars were intentionally varied. The strip from the pack with 2" (5.08 cm) wide steel bars was free of cracks, while the strip from the pack with 1" (2.54 cm) wide bars had edge cracks. There were other instances of varying steel bar widths in which the results were confounded by other variables, but those results suggested that the restraint of the steel bars was a significant factor.

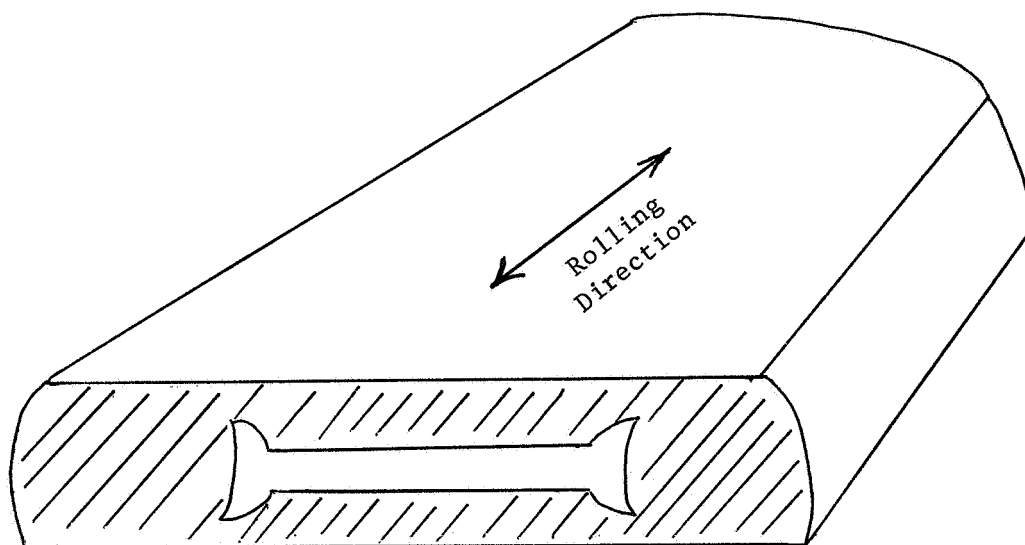
Molybdenum has a lower coefficient of thermal expansion than chromium. Therefore, as a composite of chromium and molybdenum cools, tensile stresses are generated in the chromium. The relative amounts of molybdenum and chromium or, perhaps, the absolute amount of molybdenum adjacent to the molybdenum-chromium interface, i.e., the dimension of the molybdenum in the direction perpendicular to the interface, determined whether the induced stresses caused cracking of the chromium alloy. The effect of varying amounts of molybdenum adjacent to the interface will be shown by the results of three operations. The cooling of the large extrusions, which were clad with approximately 0.06" (0.15 cm) of molybdenum, from annealing did not cause cracks. At an intermediate stage of the rolling process, the packs were cooled to ambient temperature with no evidence of cracks having been formed in the chromium alloy then. The absolute amount of molybdenum at the interface at the end of the strip had then been increased to about 0.135" (0.34 cm) by accumulation at the ends due to rolling. However, cracks formed at the ends of 1/4" plate during cooling where the original cladding was left on the extrusion. The original 0.06" (0.15 cm) cladding was increased at the ends of the plate to about 0.24" (0.61 cm) by the rolling operation. The critical amount of molybdenum for cracking lay between 0.135" (0.34 cm) and 0.24" (0.61 cm). For the 1/4" product, this type of cracking was avoided by removing nearly all of the original cladding at the ends of the extrusion before rolling.

For the 1/16" product, this type of cracking was avoided by hot shearing the ends of the strip to remove the concentration of molybdenum before the strip was cooled.

Even then, the 1/16" product may have contained significant residual stresses. Very frequently, a typical type of crack occurred during abrasive sawing of the 1/16" product into 1" x 2" coupons. These cracks extended along the surface for much greater distances than through the thickness. This behavior of the cracks could be explained by assuming that the surface of the strip had residual tensile stresses which tended to assist crack propagation, while the interior had compressive residual stresses which tended to resist crack propagation. Although a mechanism connecting the differential contraction and residual stresses is not obvious, it seems likely that they are causatively related.

The abrasive sawing operation lagged several weeks behind the rolling operations. As a result, when the possibility of adverse residual stresses in the 1/16" product was recognized, the rolling of that product had been nearly completed. However, an attempt was made to reduce thermally induced residual stresses in the 1/4" product by cooling the packs much more slowly after the final annealing treatment. Since there are, of course, several other differences between the 1/16" and 1/4" products, the absence of cracking during the abrasive sawing of the 1/4" product does not establish the change in the cooling procedure as the cause. Appendices A and B present the detailed processing parameters, including the cooling procedure, for the rolled products. Not only were the temperature gradients in the 1/4" product less than those of the 1/16" product during cooling, but also, the volume change accompanying the allotropic change of the steel undoubtedly occurred while the steel was at a higher temperature, and consequently weaker.

Jacketing caused nonuniform deformation of the chromium alloy during rolling. Since the chromium alloy was much stronger than the steel jacket at the rolling temperatures, 2192°F (1473°K) to 2372°F (1573°K), the steel could transmit sufficient pressure to the chromium alloy to deform both materials uniformly during rolling only where the steel was effectively restrained. In areas remote from the ends and edges of the chromium alloy, the steel was restrained between the cold, strong steel rolls and the relatively strong chromium alloy, so that both the chromium alloy and the steel were reduced in thickness proportionally. Near the edges and ends of the chromium alloy, however, the steel was not effectively restrained; the reduction in thickness of the chromium alloy during rolling tended to be less as shown on the following page:



Since the chromium alloy was not reduced as much in thickness at the edge as at the center, the elongation in the rolling direction tended to be less at the edge. The difference in elongation was believed to be the cause of some edge cracks, specifically in Task 1 extrusions rolled in jackets with 0.134" (0.34 cm) thick covers. Thinner steel jackets reduced the variation in the thickness of the chromium alloy and the width of the region where the thickening of the alloy was significant, but 0.060" (0.15 cm) thick jacket covers ruptured during the fifth or sixth rolling pass. So, the final procedure was to jacket with 0.06" (0.15 cm) thick covers, roll four passes, add 0.100" (0.25 cm) covers, and complete the rolling. The 1/16" product had a region at the edges about 1/8" (0.3 cm) wide in which there was a measurable increase in thickness. The thickness at the edge was typically 0.008" to 0.010" (0.02 cm to 0.025 cm) thicker than the thickness at the center.

The molybdenum cladding on the Task 2 extrusions, which was about 0.06" (0.15 cm) thick, would be reduced in thickness to about 0.004" (0.01 cm) during rolling of the 1/16" product. Since it was thought that thickness might be insufficient to prevent diffusion of iron through the molybdenum to the chromium alloy, an additional shim of molybdenum 0.050" (0.127 cm) was placed between the extrusion and the steel covers for the 1/16" product, but not for the 1/4" product. Microprobe analysis of the 1/16" product later indicated that the entire change in the measured iron concentration occurred in a zone less than 0.001" (0.0024 cm) thick at the steel-molybdenum interface. The entire change in molybdenum concentration was detected as occurring in a zone less than 0.001" (0.0024 cm) thick at the chromium alloy-molybdenum interface. The surface of the rolled products was electroetched to remove 0.002" (0.005 cm) per surface; this was more than sufficient to remove all chromium alloy enriched in molybdenum.

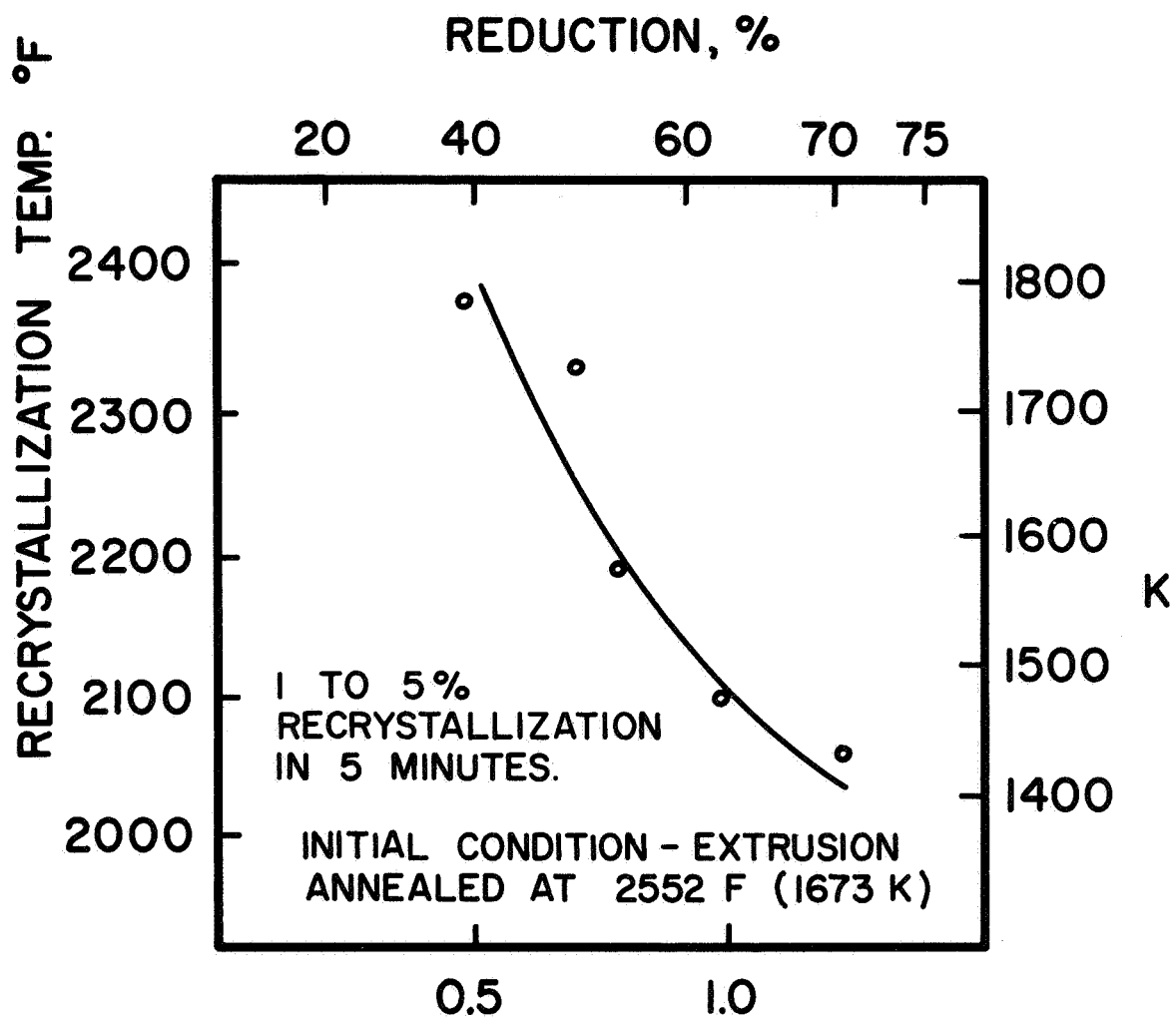
Development of Rolling and Annealing Processes - The development of the rolling and annealing practices evolved in three stages with varying approaches. The first approach was to perform only warm rolling and stress relief annealing; this was an attempt to adapt conventional refractory metal fabrication practices to this complex alloy. The next approach involved the use of in-process recrystallizations to improve fabricability and to provide better properties in the product. The third approach was an attempt to improve a property, the ductile to brittle bend transition temperature (DBTT), by minor process variations.

The initial rolling experiments involved warm working at temperatures somewhat below the temperature for the start of recrystallization (Figure 9). It was apparent, however, that the recrystallization temperature after the necessary amount of warm work would be below the temperatures that the alloy would be expected to encounter in any likely application. Thus, the beneficial effects of warm working would be lost. Therefore, this approach for the development of a rolling process was abandoned.

The next general scheme of rolling involved recrystallizing the alloy after about 63% warm work to reduce the grain size and to make the grain size uniform. This was followed by warm rolling, a second recrystallization anneal, and more warm work. The purpose of the second recrystallization was to limit the warm work in the product so that its recrystallization temperature would be relatively high and some beneficial effects of warm work might survive exposure at anticipated service temperatures.

The conditions of time and temperature for the recrystallization anneals were established on the basis of a cursory metallographic study and other considerations. The upper limit for temperature was dictated by the possibility of melting at the molybdenum-carbon steel interface of the jacket. No evidence of melting was observed in packs annealed at 2462°F (1623°K). This temperature provided an adequate margin above the minimum temperature for complete recrystallization in 15 minutes, which was estimated to be 2372°F (1572°K) for the alloy with 60% or more warm work from the annealed extrusion condition. Annealing at 2462°F (1623°K) for 15 minutes was chosen as the standard recrystallization treatment.

The prototype of the process with two recrystallizations was the process designated as Process B. The essentials of this process and all subsequent developmental and production processes are presented graphically in Figures 10 through 13. Process B was warm rolling to a reduction of 63% at temperatures decreasing from 2372°F (1573°K) to 2057°F (1398°K), recrystallizing, warm rolling to an additional reduction of 63% at decreasing temperatures, recrystallizing, warm rolling to a reduction of 58%, and annealing. In Process B, as well as all later developmental processes and the Task 2 processes, the packs were stored in an auxiliary furnace at 1562°F (1123°K) to 1652°F (1173°K) while temperature changes were being made in the furnace used for rolling and annealing. It was believed that these temperatures would have no effect on the properties or structure, and yet they would exceed the transition temperature so that cracking by differential thermal contraction was avoided.



## TRUE STRAIN DURING WARM ROLLING

Figure 9 Recrystallization Temperature

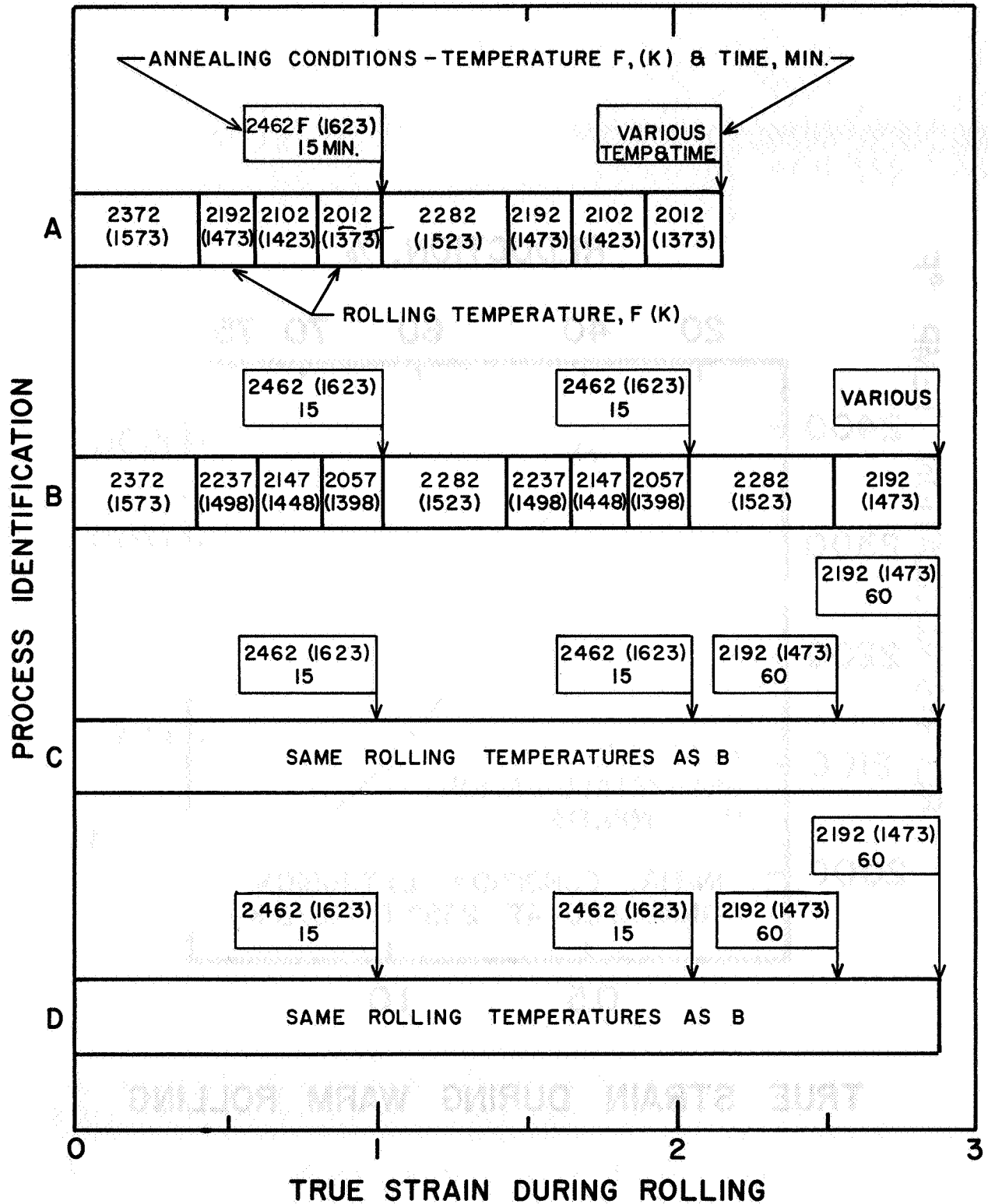


Figure 10 Rolling and Annealing Processes

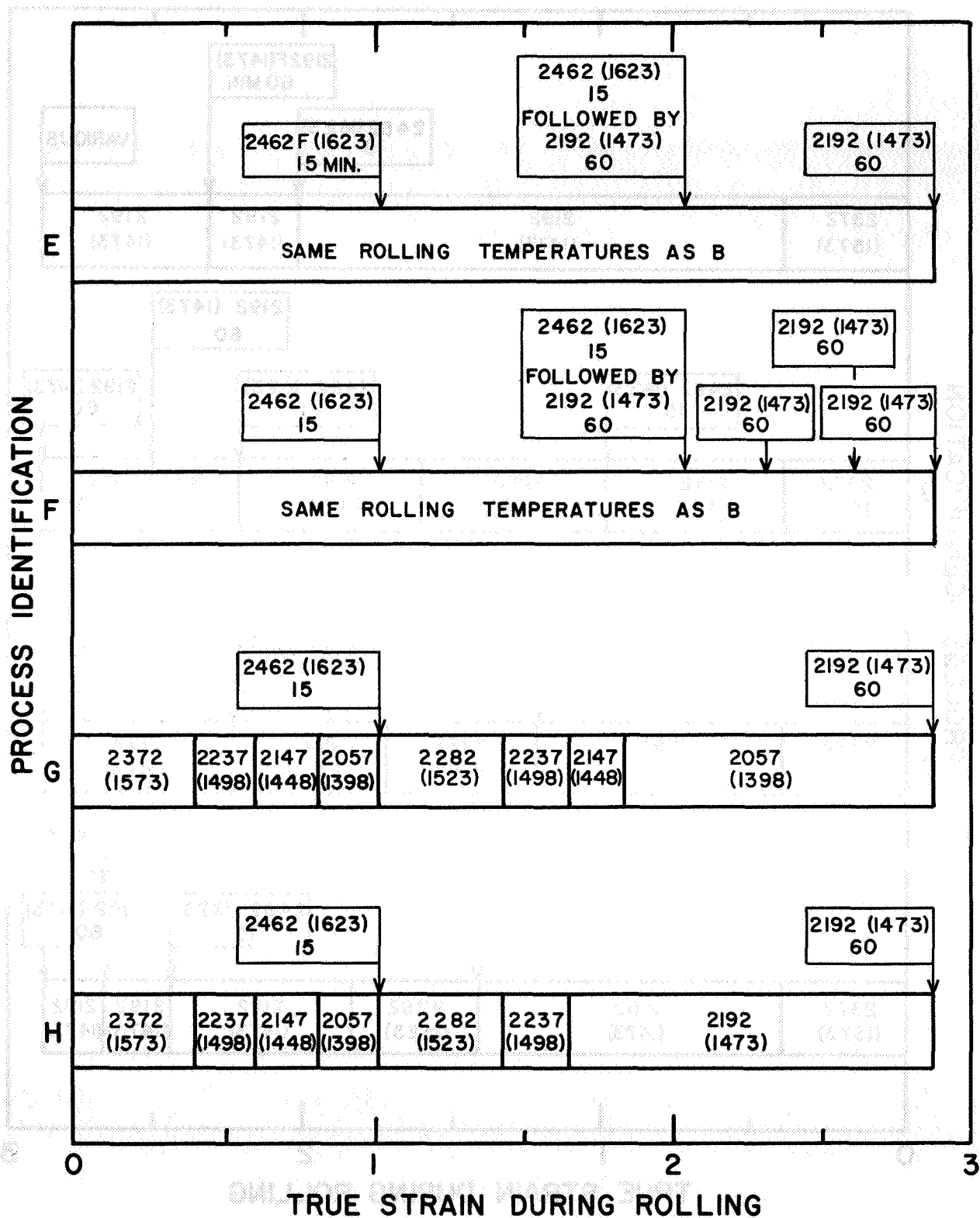


Figure 11 Rolling and Annealing Processes

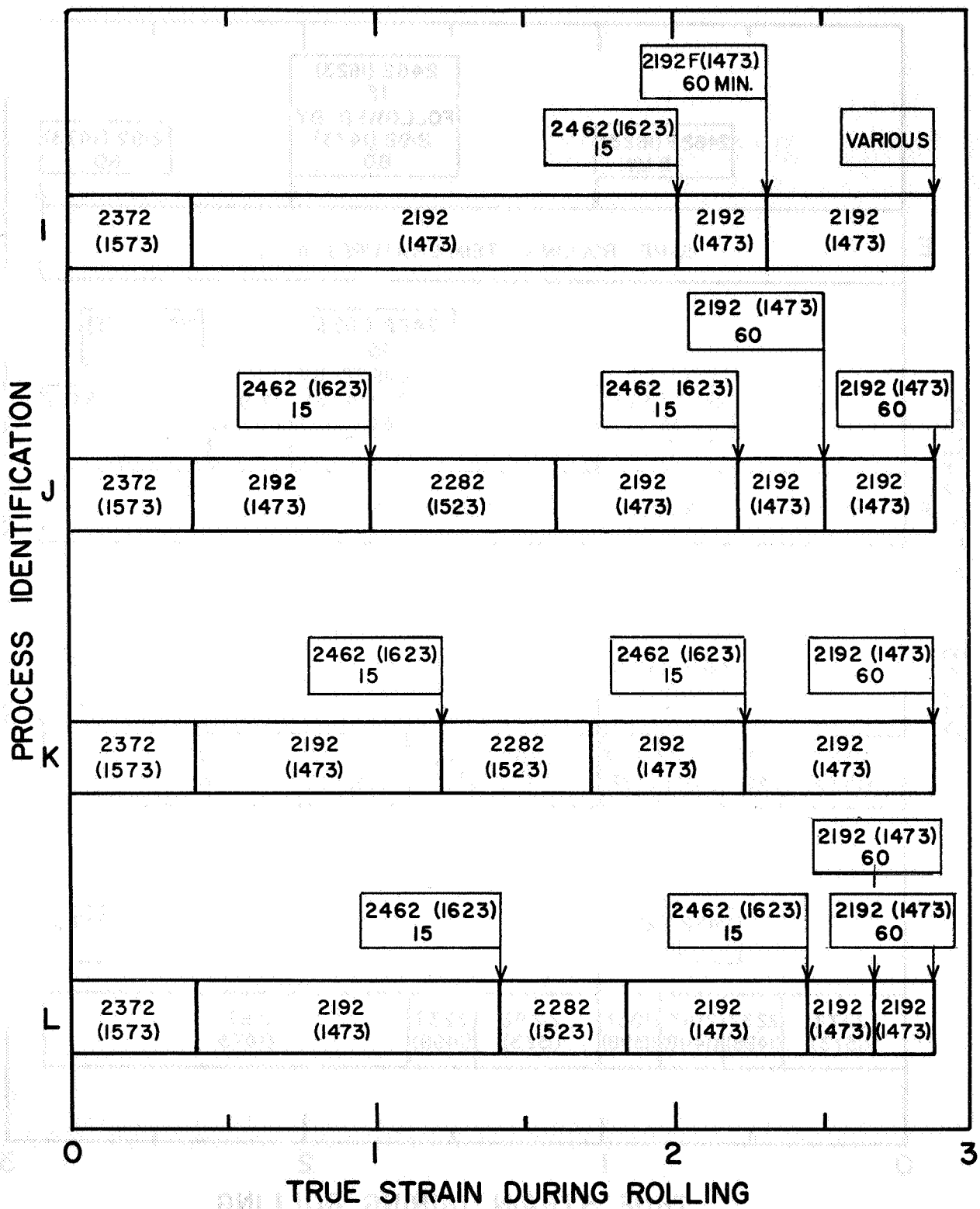


Figure 12 Rolling and Annealing Processes



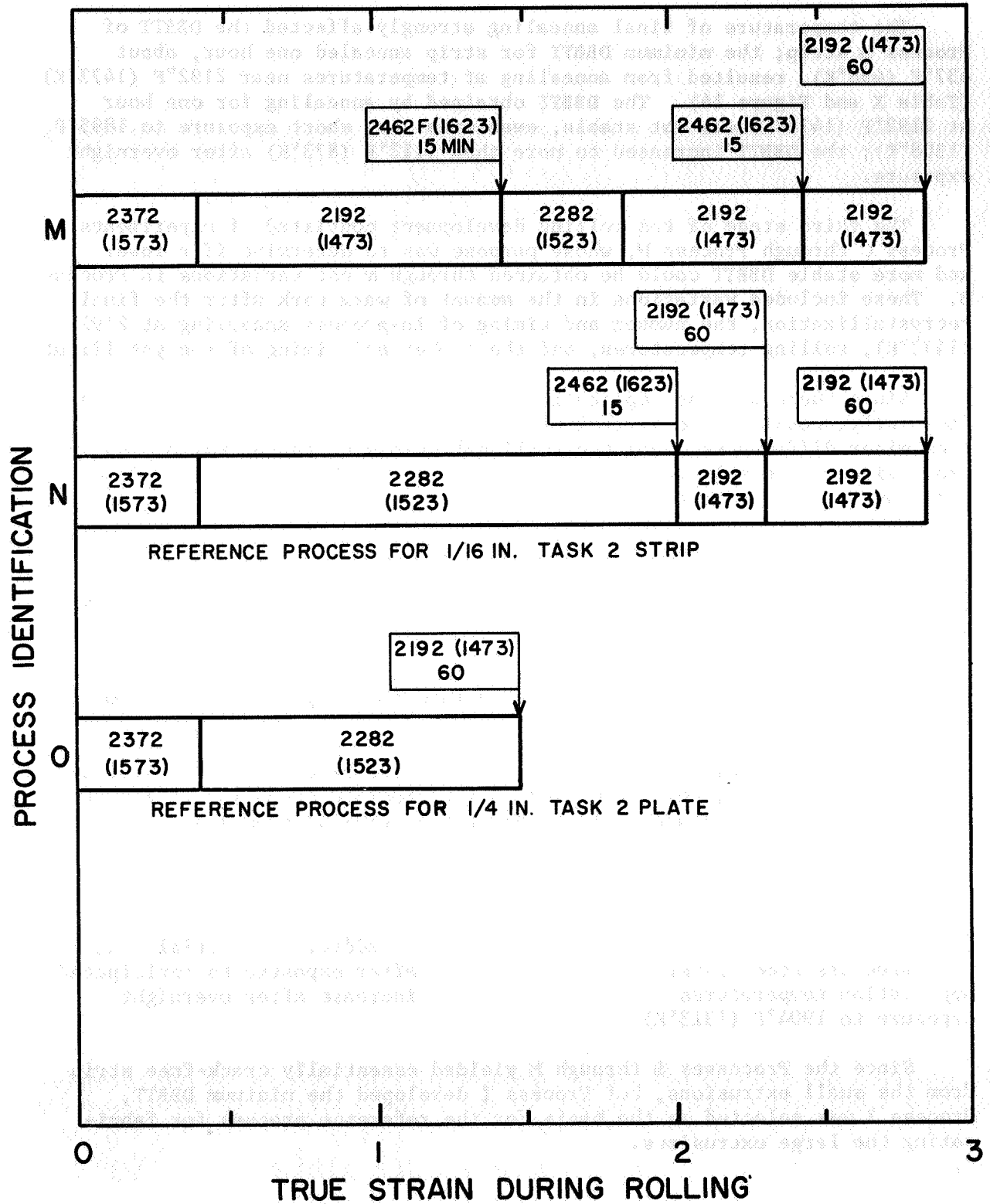


Figure 13 Rolling and Annealing Processes

The temperature of final annealing strongly affected the DBBTT of Process B strip; the minimum DBBTT for strip annealed one hour, about 437°F (498°K), resulted from annealing at temperatures near 2192°F (1473°K) (Table X and Figure 14). The DBBTT obtained by annealing for one hour at 2192°F (1473°K) was not stable, even with very short exposure to 1895°F (1308°K); the DBBTT increased to more than 1112°F (873°K) after overnight exposure.

The third stage of the rolling development consisted of experiments. Process C through Process M, whose purpose was to determine if a lower and more stable DBBTT could be obtained through minor variations in Process B. These included variations in the amount of warm work after the final recrystallization, the number and timing of in-process annealing at 2192°F (1473°K), rolling temperatures, and the number and timing of recrystallizations.

Since there were no replications of these experiments, and there was, apparently, considerable variation in the DBBTT of Process B strip resulting from minor differences among the small extrusions or random variations in processing, the conclusions drawn from the results have to be considered semiquantitative at best.

1. In-process annealing at 2192°F (1473°K) affected the DBBTT.
2. Annealing at 2192°F (1473°K) immediately after the final recrystallization increased the DBBTT.
3. Annealing after the final recrystallization and one or two subsequent warm rolling passes, but before the completion of the rolling, decreased the DBBTT.
4. Warm working to a total reduction of 58% after the final recrystallization, which included an intermediate annealing at 2192°F (1473°K), produced a lower DBBTT than similar processes which involved warm working to reductions of 44% or 26%.
5. Final annealing at 2192°F (1473°K) also decreased the DBBTT.

Process I developed the minimum DBBTT. In addition, material thus processed has some resistance to degradation after exposure to anticipated application temperatures. The DBBTT did not increase after overnight exposure to 1904°F (1313°K).

Since the Processes B through M yielded essentially crack-free strip from the small extrusions, but Process I developed the minimum DBBTT, Process I was selected as the basis for the reference process for fabricating the large extrusions.

TABLE X. - DUCTILE TO BRITTLE BEND TRANSITION TEMPERATURES  
OF STRIP AFTER PROCESSING

Process identification (a)	Extrusion number	Final annealing conditions		Transition temperature	
		Temperature F	Time, minutes	Temperature F	Temperature K
A	996-30T	1832	60	887	748
		1967	60	797	698
B	994-30B	2012	50	590<DBBTT<932	583<DBBTT<773
		2192	4080	>1112	>873
		As rolled			
		1832	60	>1112	>873
		2012	60	>1076	>853
				752<DBBTT<932	673<DBBTT<773
		2084	60	<527	<548
		2102	60	482	523
		2192	60	<482	<523
		2102	240	437	498
	995-30T	As rolled			
		2192	30	977	798
		2057	60	617	598
				572	573
		2102	60	572	573
		2147	60	527	548
		2192	60	437	498
		2237	60	437	498
		2282	60	662	623
		2327	60	752<DBBTT<842	673<DBBTT<723
		2417	60	752	673
		1895	900	>842	>723

TABLE X. - DUCTILE TO BRITTLE BEND TRANSITION TEMPERATURES OF STRIP  
AFTER PROCESSING (Continued)

Process identification.	Extrusion number	Final annealing conditions			Transition temperature	
		F	Temperature	Time, minutes	F	K
B	995-30T	2012	1373	960	>842	>723
		2102	1423	900	>1112	>873
	Sequential anneals followed by	2192	1473	60		
		1895	1308	900	>1112	>873
C	996-30B	As rolled	1473	60	842<DBBTT<932	723<DBBTT<773
		2192			662	623
		As rolled	1473	60	>1112	>873
		2192			482	523
D	996-30B	As rolled	1473	60	>1112	>873
		2192			437	498
		As rolled	1473	60	842<DBBTT<932	723<DBBTT<773
		2192			752	673
E	996-30B	As rolled	1473	60	>1112	>873
		2192			527	548
		As rolled	1473	60	752<DBBTT<1112	673<DBBTT<873
		2192			872<DBBTT<932	723<DBBTT<773
F	996-30B	As rolled	1473	60	>1112	>873
		2192			527	548
		As rolled	1473	60	752<DBBTT<1112	673<DBBTT<873
		2192			872<DBBTT<932	723<DBBTT<773
G	996-30B	As rolled	1473	60	>1112	>873
		2192			527	548
		As rolled	1473	60	752<DBBTT<1112	673<DBBTT<873
		2192			872<DBBTT<932	723<DBBTT<773

TABLE X. - DUCTILE TO BRITTLE BEND TRANSITION TEMPERATURES OF STRIP  
AFTER PROCESSING (Continued)

Process identification	Extrusion number	Final annealing conditions			Transition temperature	
		F	Temperature K	Time, minutes	F	K
H	996-30B	As rolled 2192	1473	60	<752 752<DBTT<842	<673 673<DBTT<723
I ↓	995-30B  Sequential anneals followed by	2192	1473	60	365 437	458 498 (c)
		2192	1473	60		
		2192 1904	1473 1313	60 960		
J	995-30B	2192	1473	60	707	648
K	995-30B	2192	1473	60	887	748
L	995-30B	2192	1473	60	842	732
M	995-30B	2192	1473	60	1067	848
N ↓	131-100  Sequential anneals followed by	2192	1473	60	554	563
		2192 2462	1473 1623	60 60		
					>1112	>873

TABLE X. - DUCTILE TO BRITTLE BEND TRANSITION TEMPERATURES OF STRIP  
AFTER PROCESSING (Continued)

Process identification	Extrusion number	Final annealing conditions			Transition temperature	
		Temperature		Time, minutes	F	K
		F	K			
N ↓	138-100	2192	1473	60	590	583
	140-100	2192	1473	60	548	560

<sup>a</sup>See Figures 10 through 13

<sup>b</sup>The ductile to brittle bend transition temperature was defined as the minimum temperature at which a 90° bend could be made without cracking. The radius at the tip of the punch was approximately four times the specimen thickness and the punch speed was 1" per minute (0.040 m/s). All tests were made with the bend axis perpendicular to the rolling direction. For the 900 series of extrusions, the transition temperature was determined to an accuracy of about 45F (25K) unless otherwise noted. For the 100 series extrusions (Task 2), the temperature was determined to an accuracy of about 18°F (10°K) according to the procedure specified in MAB-192-M "Evaluation Test Methods for Refractory Metal Sheet Material", dated April 22, 1963.

<sup>c</sup>Replication of the final annealing and testing operations.

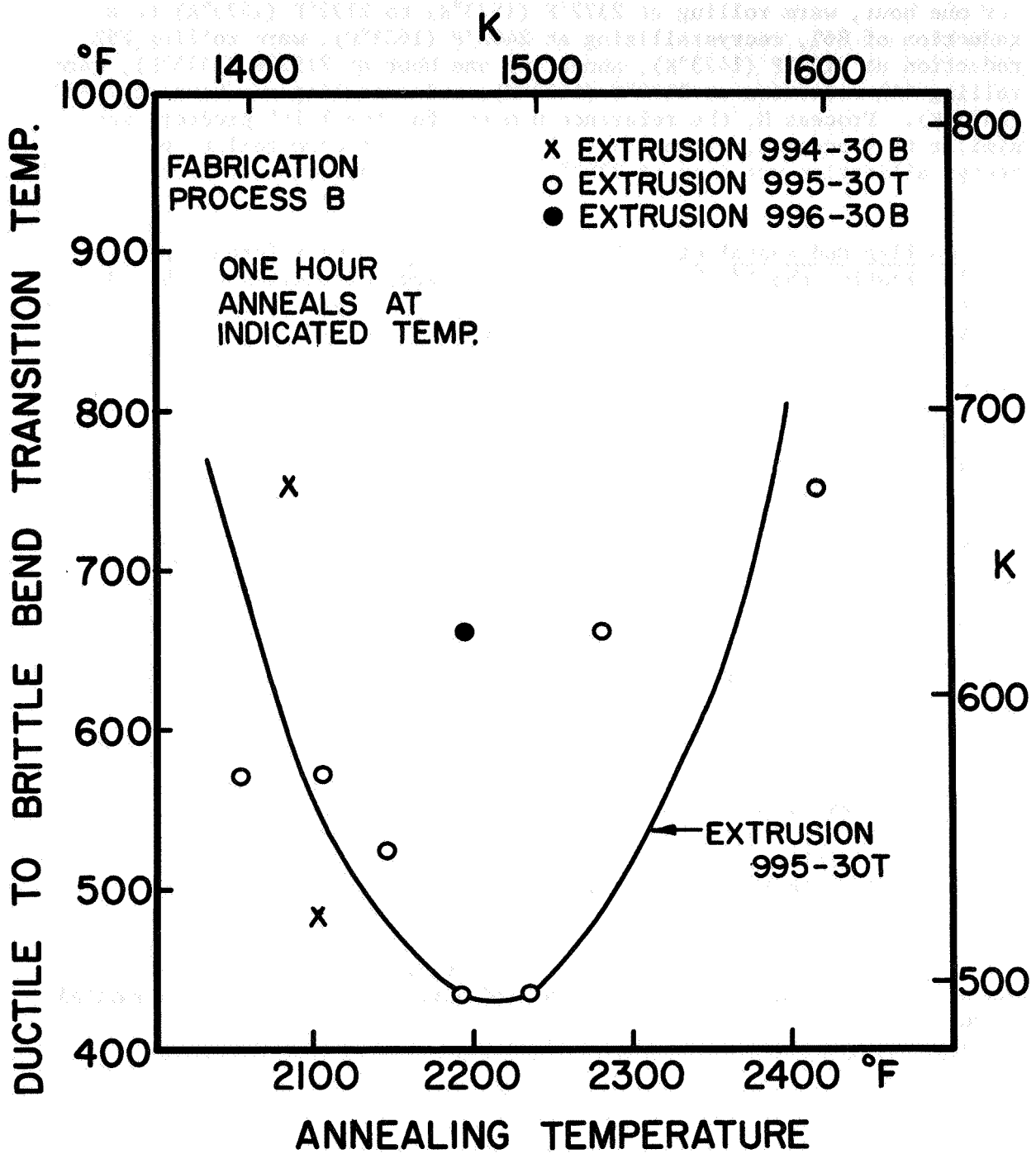


Figure 14 Ductile to Brittle Bend Transition Temperature

Process I consisted of annealing the extrusion at 2552°F (1673°K) for one hour, warm rolling at 2372°F (1573°K) to 2192°F (1473°K) to a reduction of 86%, recrystallizing at 2462°F (1623°K), warm rolling 25% reduction at 2192°F (1473°K), annealing one hour at 2192°F (1473°K), warm rolling 44% reduction at 2192°F (1473°K), and annealing one hour at 2192°F (1473°K). Process N, the reference process for the 1/16" product, was similar to Process I, except that the increment of warm rolling prior to recrystallization was done at 2282°F (1523°K) rather than at 2192°F (1473°K).

Rolling and Annealing of the 1/16" Product - The reference process for fabricating the 1/16" product from the large extrusions was based on Process I of the development phase with minor changes introduced to improve the yield. Because of numerous cracks in the large extrusions, the cause of cracks in the product could not be readily associated with the rolling process. But, as the rolling progressed, it became apparent that the rolling process was contributing to edge cracking. Although the chromium was jacketed, indications of the cracking of the alloy could be seen occasionally as the packs emerged from the rolling mill. The thermal properties of the pack were such that the different surface temperature color indicated where open cracks existed in the alloy. When it was established that cracking was occurring often during the intermediate stages of rolling, the temperature was increased to 2282°F (1523°K) for that part of the process. A definite improvement in yield resulted. A detailed description of the final process is presented in Figure 13 and Appendix A. The deviations from the final process, which occurred in the early rolling lots, are described in Appendix C.

Rolling and Annealing of the 1/4" Product - Since there was to be no testing of the 1/4" product for mechanical properties, the only criterion for success was yield. Therefore, the 1/4" product was rolled in the same manner as the 1/16" product with the appropriate reduction. The final annealing conditions, 2192°F (1473°K) for one hour, were chosen arbitrarily. A detailed description of the process is presented in Figure 13 and Appendix B.

Processing of Other Products - The other products, 3/8" x 3/8" x 3" and 3/4" x 1-1/2" x 2", were machined and abrasive sawed from an annealed extrusion.



## EVALUATION OF PRODUCTS

Mechanical Properties - The 1/16" (0.16 cm) thick product was tested both as fabricated and after annealing at 2462°F (1623°K) for one hour. The mean DBBT of the as-fabricated strip was 564°F (569°K), but annealing at 2462°F (1623°K) increased the DBBT to more than 1112°F (873°K), the upper limit for testing (Table X). The short-time strength at elevated temperatures was increased substantially by the annealing (Figures 5 to 7 and Table IX).

The mechanical properties were not determined for products which were machined from the annealed large extrusion, but short time elevated temperature tensile properties were determined for a small extrusion which had a similar annealing treatment, Table IX. Mechanical properties of the 1/4" (0.59 cm) plate were not determined.

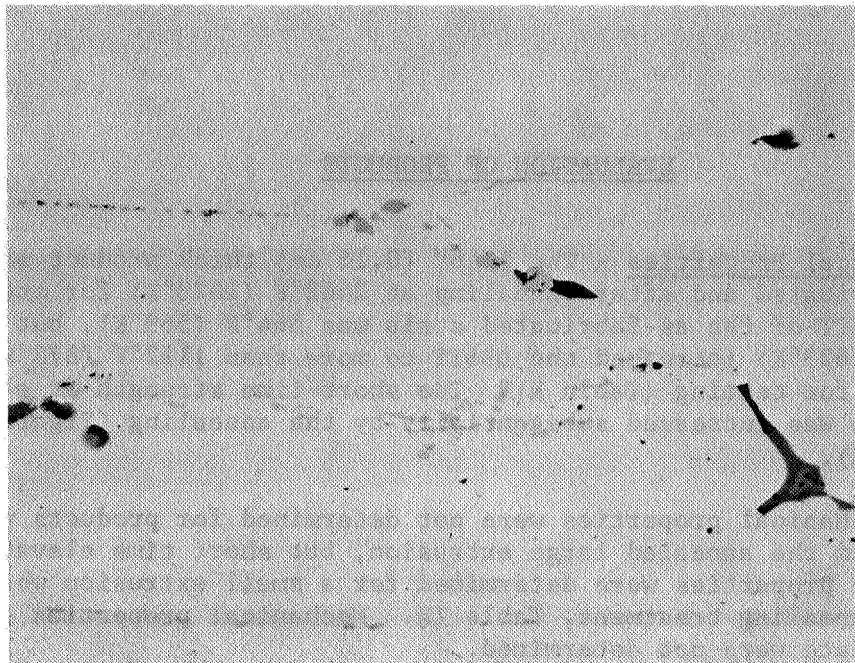
Dimensions and Finish - There were only minor and infrequent deviations from the requirements for dimensions and finish. The thickness tolerance was plus or minus 4% of the nominal thickness. The finish met the requirement of 80 microinch AA for products up to 3/8" (0.95 cm) thick.

Cracks - After being cut to size, only the 1/16" coupons had significant cracking. The coupons had cracks or crack-like defects of two different origins and types. Apparently, some cracks in the extrusions were bonded to a greater or lesser extent during rolling. These appeared in the product as offsets in the surface and as intermittent longitudinal cracks in, perhaps, 1% or 2% of the coupons.

Other cracks originated during the abrasive cutting operation. These cracks were believed to be caused by residual stresses (as discussed in an earlier section) and were characterized by their being shallow in relation to their length. Their frequency varied greatly from lot to lot. The best lots were nearly free of this defect, while the worst lots had approximately one-third of their coupons affected.

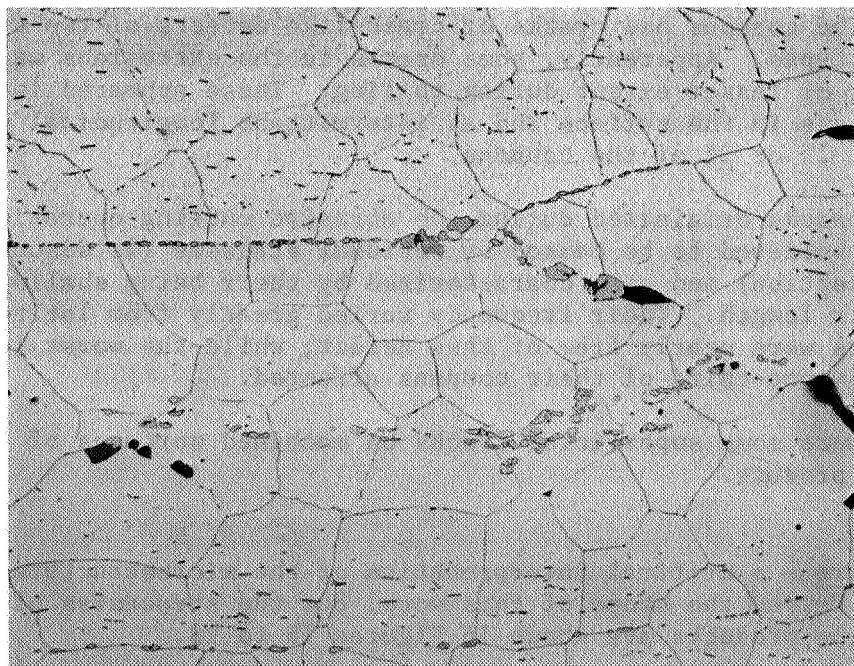
There were some superficial cracks on machined surfaces of the 3/4" x 1-1/2" x 2" product.

Structure - a critical interpretation of the microstructures of the products is beyond the scope of this work, but the structures of random samples of the products are shown in Figures 15 to 18.



Unetched

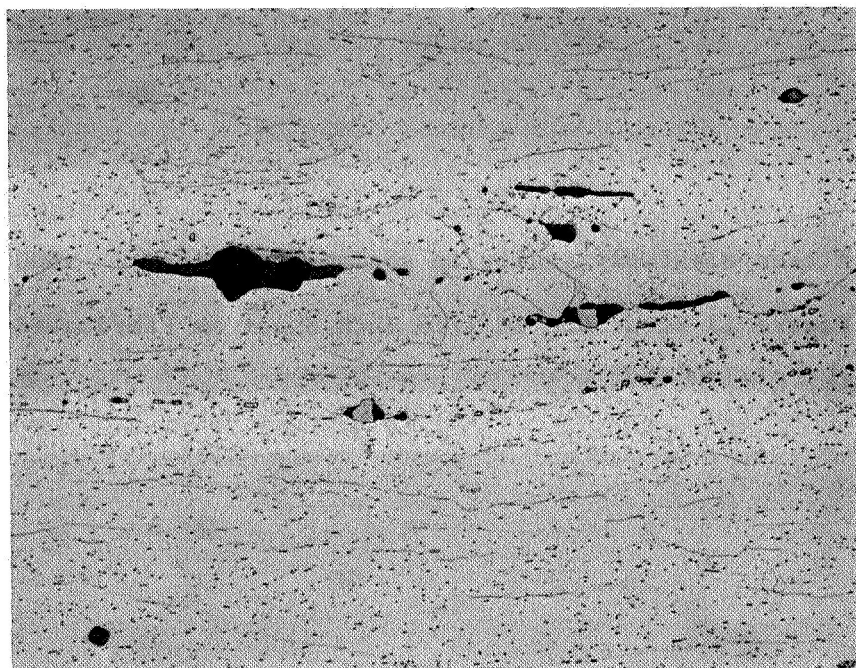
x500



Etched electrolytically in sodium hydroxide

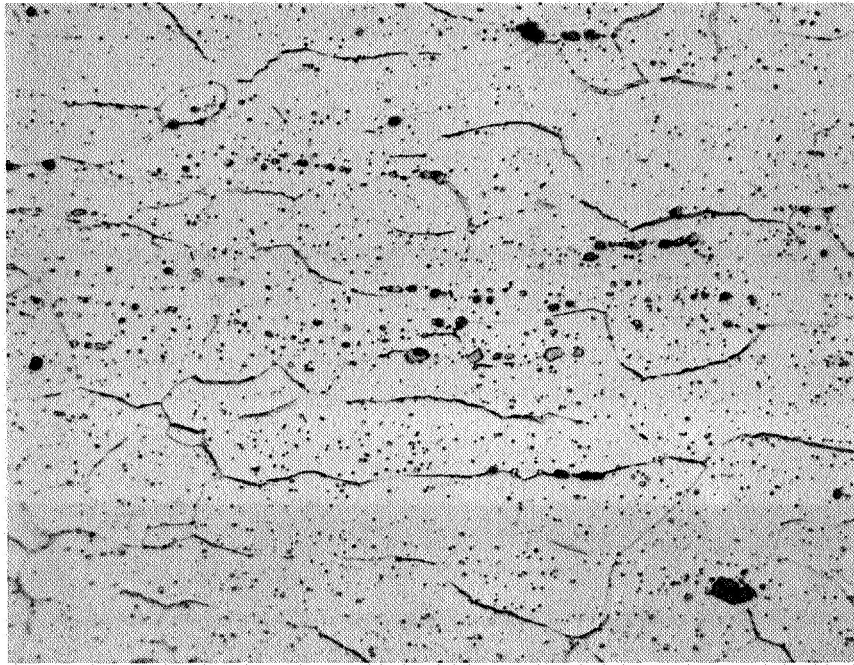
x500

Figure 15 Structure of Annealed Extrusion from 100 Pound Heat



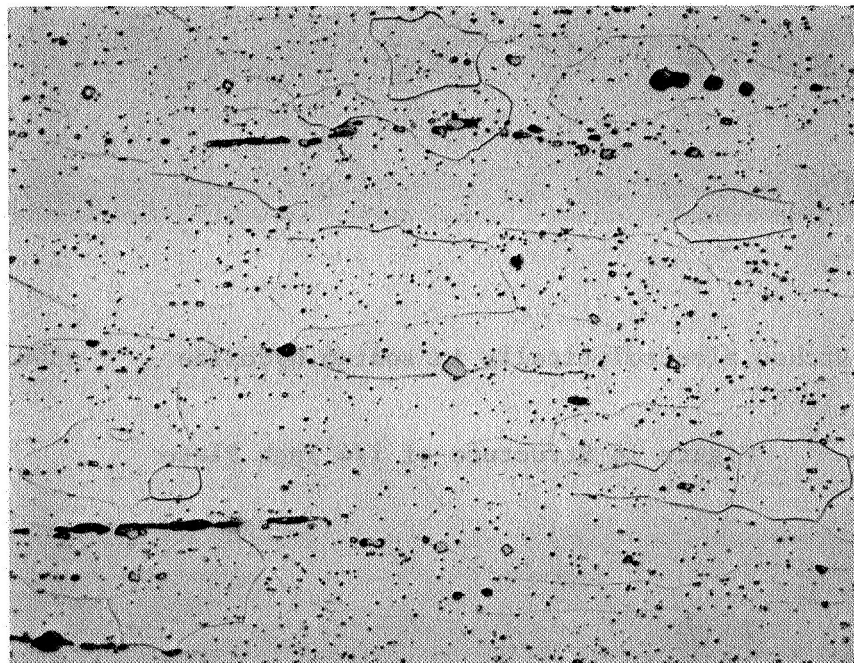
Etched electrolytically in sodium hydroxide      x500

Figure 16 Structure of 1/4 Inch Plate



Etched electrolytically in sodium hydroxide x500

Figure 17 Structure of 1/16 Inch Sheet as Fabricated



Etched electrolytically in sodium hydroxide x500

Figure 18 Structure of 1/16 Inch Sheet Annealed at 2462F(1623K)

Composition - Analyses for selected elements were made on products from each of the large heats. The results are shown in Table VII. The apparent decrease in the oxygen and nitrogen contents from the ingot to product was unexpected; therefore, an attempt was made to resolve the contradiction by repeating both the ingot and product analyses for representative heats. The duplicate analyses for the product confirmed the original analyses within the expected error. Unfortunately, there was a malfunction of the vacuum fusion apparatus so that valid analyses for the ingots were not obtained.

Yield - The yield of product from the 100 pound (45.36 kg) melts was as shown in this table:

<u>Product Size, in.</u>	<u>Approximate Weight per Piece, lbs</u>	<u>Approximate Total Weight, lbs</u>
1/16 x 1 x 2	0.03	39
1/4 x 1 x 4	0.26	23
3/8 x 3/8 x 3	0.11	6
3/4 x 1-1/2 x 2	0.59	18

APPENDIX A. - DETAILED PROCESS DESCRIPTION  
FOR SECONDARY FABRICATION OF CHROMIUM ALLOY SHEET

<u>Operation no.</u>	<u>Operation</u>	<u>Description</u>
1	Annealing of extrusion	The chromium alloy extrusion was preheated to 1651°F (1173°K). This operation, as well as all other heating operations, was done in a hydrogen atmosphere. The extrusion was then transferred to the annealing furnace and annealed for one hour at 2552°F (1673°K). The extrusion was next returned to the furnace at 1652°F (1173°K) and allowed to cool to that temperature. Finally, the extrusion was cooled to ambient temperature in a hydrogen atmosphere in about 30 minutes.
2	Jacketing of extrusion	The extrusion was band sawed and machined into sections approximately 2.38" (6 cm) long. The exposed chromium alloy was wet ground with 180 grit abrasive paper and electroetched. The exposed alloy was inspected for cracks by visual or fluorescent penetrant inspection. The jacket was then assembled around the extrusion and welded.
3	Rolling	The pack, i.e., the assembly of the extrusion and its jacket, was evacuated for 15 minutes by a mechanical vacuum pump and the evacuation tube was sealed. The pack was preheated for 30 minutes at 1652°F (1173°K) and then was heated for 20 minutes before the first rolling pass and for lesser lengths of time before later rolling passes. The minimum heating time was five minutes. The pack was warm rolled the first two rolling passes at 2372°F (1573°K) to a reduction of 18% per pass at a mill speed of 40 ft./min. (0.203 m/s). The diameter of the rolls was 12" (30.5 cm). After the first two rolling passes, the pack was transferred to a furnace at 1652°F (1173°K) so that the temperature of the rolling mill furnace could be changed. The pack was then rolled two passes at 18%

APPENDIX A. - DETAILED PROCESS DESCRIPTION FOR  
SECONDARY FABRICATION OF CHROMIUM ALLOY SHEET (Continued)

<u>Operation no.</u>	<u>Operation</u>	<u>Description</u>
		reduction each at 2282°F (1523°K). The pack was annealed at 1562°F (1173°K) for 30 minutes, furnace cooled to approximately 1292°F (973°K) and, finally, cooled to ambient temperature buried in insulating material.
		A rolling lot consisted of from one to nine packs assembled at one time. No more than five packs could be accommodated in the two furnaces used for:
		a. Preheating and storing at 1652°F (1173°K) b. Heating for rolling and for annealing
		Thus, rolling and annealing was accomplished on groups of no more than five packs. Packs were heated for rolling individually, while the rest of the group was stored at 1652°F (1173°K). No more than two packs were annealed simultaneously at 2462°F (1623°K).
4	Replenishment of jacketing	The pack was grit blasted and ground to remove scale. An additional sheet of steel, 0.10" (0.25 cm) thick, was welded to the top and to the bottom of the pack.
5	Rolling	After being preheated for 20 minutes at 1652°F (1173°K), the pack was rolled six passes of 18% reduction each at 2282°F (1523°K) with a mill speed of 40 ft./min. (0.203 m/s). Excess steel at the ends of the pack was removed by hot shearing and the pack stored briefly at 1652°F (1173°K).
6	Annealing	The pack was annealed at 2462°F (1623°K) for 15 minutes and stored at 1652°F (1173°K) while the rolling mill furnace temperature was being changed.

APPENDIX A. - DETAILED PROCESS DESCRIPTION FOR  
SECONDARY FABRICATION OF CHROMIUM ALLOY SHEET (Continued)

<u>Operation no.</u>	<u>Operation</u>	<u>Description</u>
7	Rolling	The pack was rolled one pass of 25% reduction at 2192°F (1473°K) at a mill speed of 80 ft./min. (0.406 m/s) and sheared into two pieces at 2192°F (1473°K).
8	Annealing	The pack was annealed one hour at 2192°F (1473°K).
9	Rolling	The pack was rolled to finish gage in two passes of approximately 25% each at 2192°F (1473°K) with a mill speed of 80 ft./min. (0.406 m/s). The ends of the pack were sheared at 2192°F (1473°K) to expose the chromium alloy. The steel at the sides of the pack was sheared within 1/8" (0.3 cm) of the chromium strip.
10	Annealing	The pack was annealed one hour at 2192°F (1473°K), held briefly in a furnace at 1652°F (1173°K) and cooled to ambient temperature in a hydrogen atmosphere.
11	Removal of jacket	The pack was pickled in a mixture of nitric, sulphuric and hydrofluoric acids to remove the steel and molybdenum jacket.
12	Electroetching	The chromium alloy strip was etched anodically in a saturated solution of oxalic acid in water to remove a layer 0.002" (0.005 cm) thick from each surface.
13	Abrasive sawing	The chromium alloy strip was clamped for sawing using the minimum force required. Shims were inserted under the strip as needed to support cambered strip. The strips were abrasive sawed using a "soft" abrasive wheel and 1/2 in./min. (0.002 m/s) feed rate.
14	Electroetching	The alloy was etched anodically for about 15 seconds.



APPENDIX A. - DETAILED PROCESS DESCRIPTION FOR  
SECONDARY FABRICATION OF CHROMIUM ALLOY SHEET (Continued)

<u>Operation no.</u>	<u>Operation</u>	<u>Description</u>
15	Inspection	The pieces were inspected at low magnification for cracks or surface defects and graded as Grade A, B, or C. Grade A pieces contained no visible defects. Grade B contained minor defects that were not considered to be serious for their use in coating studies. Grade C pieces contained serious defects.

APPENDIX B. - DETAILED PROCESS  
DESCRIPTION FOR SECONDARY FABRICATION OF 1/4" PLATE

<u>Operation no.</u>	<u>Operation</u>	<u>Description</u>
1 through 4	These operations were the same as those for 1/16" strip.	
5	Rolling	After being preheated for 20 minutes at 1652°F (1173°K), the pack was rolled to gage in three passes of about 19% reduction each at 2282°F (1523°K) at a mill speed of 40 ft./min. (0.203 m/s). The pack was stored briefly at 1652°F (1173°K) while a furnace temperature change was being made.
6	Annealing	The pack was annealed one hour at 2192°F (1473°K), then held in a furnace at 1652°F (1173°K) for 30 minutes, furnace cooled to approximately 1292°F (973°K) and, finally, cooled to ambient temperature buried in insulating material.
7	Removal of jacket	Excess steel was sawed from the ends and sides of the pack. The pack was pickled in a mixture of nitric, sulphuric and hydrofluoric acids to remove the steel and molybdenum jacket.
8	Electroetching	The chromium alloy plate was etched anodically in a saturated solution of oxalic acid in water to remove a layer of the alloy, 0.002" (0.005 cm) thick, from each surface.
9	Abrasive sawing	The chromium alloy plate was abrasive sawed to the required size, 1/4" x 1" x 4". (0.61 cm x 2.54 cm x 10.16 cm) by plunge cutting in two or three passes.
10	Electroetching	The alloy was electroetched for about 15 seconds.
11	Inspection	The pieces were inspected at low magnification for surface defects.

APPENDIX C. - DEVIATIONS FROM STANDARD PROCESSES

<u>Lot number</u>	<u>Deviations</u>
37	First rolling pass mill speed was 100 fpm (0.508 m/s), mill stalled. The third through tenth rolling pass temperature was 2192°F (1473°K). At the conclusion of Operation 3, the pack was cooled from 1652°F (1173°K) to ambient temperature in a hydrogen atmosphere.
38	The third through tenth rolling pass temperature was 2192°F (1473°K). At the conclusion of Operation 3, the pack was cooled from 1652°F (1173°K) to ambient temperature in a hydrogen atmosphere.
39	Same deviations as Lot 38
40	Same deviations as Lot 38
42	Mill stalled during the first pass because the chromium was wider than standard. The third through tenth rolling pass temperature was 2192°F (1473°K). At the conclusion of Operation 3, the pack was cooled from 1652°F (1173°K) to ambient temperature in a hydrogen atmosphere.
43	Same deviations as Lot 38
44	Same deviations as Lot 38
45	Same deviations as Lot 38
46	The third through tenth rolling pass temperature was 2192°F (1473°K).
47	Same deviation as Lot 46
48	Same deviation as Lot 46
48A	Third and subsequent rolling passes temperature was 2192°F (1473°K). Pack was cooled to ambient temperature before the final anneal.
49	No deviations
50	No deviations
51	No deviations

APPENDIX C. - DEVIATIONS FROM STANDARD PROCESSES (Continued)

<u>Lot number</u>	<u>Deviations</u>
52	No deviations
53	No deviations
54	No deviations
55	No deviations

NOTE: See Appendix D. for the correlation of heats and rolling lots for end uses.

APPENDIX D. - END USES OF THE 100 POUND HEAT EXTRUSIONS

<u>Extrusion number</u>	<u>Rolling lot number</u>	<u>Product size, inches</u>
131-100	37	1/16 x 1 x 2
	38	1/16 x 1 x 2
	39	1/16 x 1 x 2
	40	1/16 x 1 x 2
	55	1/16 x 1 x 2
138-100	42	1/16 x 1 x 2
	43	1/16 x 1 x 2
	44	1/16 x 1 x 2
	45	1/16 x 1 x 2
	46	1/16 x 1 x 2
	47	1/16 x 1 x 2
139-100	Not rolled, product cut from annealed extrusion	3/8 x 3/8 x 3
		3/4 x 1-1/2 x 2
140-100	48	1/16 x 1 x 2
	48A	1/4 x 1 x 4
	49	1/16 x 1 x 2
	54	1/4 x 1 x 4
141-100	50	1/4 x 1 x 4
	51	1/4 x 1 x 4
	52	1/4 x 1 x 4
	53	1/4 x 1 x 4



#### REFERENCES

- <sup>1</sup>Goetz, L. J., Hughes, J. R., and Moore, W. F., "The Pilot Production and Evaluation of Chromium Alloy Sheet and Plate" NASA CR-72184, March 15, 1967





## DISTRIBUTION LIST

(The number in parentheses is the number of copies sent to each addressee)

NASA Headquarters  
600 Independence Avenue  
Washington, D. C. 20546  
Attention: RAP/N. F. Rekos (1)  
RRM/G. C. Deutch (1)  
RRM/R. H. Raring (1)

NASA-Lewis Research Center  
21000 Brookpark Road  
Cleveland, Ohio 44135  
Attention: 105-1/G. M. Ault (1)  
3-19 Technology Utili-  
zation Office (1)  
105-1/William Klopp (2)  
105-1/N. T. Saunders (1)  
60-3/Library (2)  
5-5/Report Control  
Office (1)  
106-2/A. E. Anglin (1)  
77-3/John H. Deford (1)  
106-2/J. Merutka (10)  
49-1/R. H. Kemp (1)  
49-1/J. W. Weeton (1)  
105-1/R. W. Hall (1)  
60-4/J. Howard Childs (1)

NASA-Ames Research Center  
Moffett Field, California 94035  
Attention: Library (1)

NASA-Flight Research Center  
P. O. Box 273  
Edwards, California 93523  
Attention: Library (1)

NASA-Goddard Space Flight Center  
Greenbelt, Maryland 20771  
Attention: Library (1)  
714/D. F. Hasson (1)  
249.1/C. E. Vest (1)

Jet Propulsion Laboratory  
4800 Oak Grove Drive  
Pasadena, California 91102  
Attention: Library (1)

NASA-Lewis Research Center  
21000 Brookpark Road  
Cleveland, Ohio 44135  
Attention: 49-1/J. C. Freche (1)  
49-1/H. B. Probst

NASA-Langley Research Center  
Langley Field, Virginia 23365  
Attention: Library (1)  
Mr. Richard Pride (1)

NASA-Manned Space Flight Center  
Houston, Texas 77058  
Attention: Library (1)  
N. Chafee E.B.4 (1)

NASA-Marshall Space Flight Center  
Huntsville, Alabama 35812  
Attention: Library (1)

NASA Scientific and Technical  
Information Facility (6)  
P. O. Box 33  
College Park, Maryland 20740

Air Force Office of Scientific Res.  
Propulsion Research Division  
USAF Washington, D. C. 20525  
Attention: Dr. M. Slawsky (1)

Bureau of Naval Weapons  
Department of the Navy  
Washington, D. C. 20525  
Attention: RRMA-2/T. F. Kearns, Chief (1)

Defense Documentation Center (DDC)  
Cameron Station (1)  
5010 Duke Street  
Alexandria, Virginia 22314

FAA Headquarters  
800 Independence Avenue, S.W.  
Washington, D. C. 20553  
Attention: Brig. Gen. J. C. Maxwell (1)  
SS/210/F. B. Howard (1)

429/Department of the Navy, ONR  
Washington, D. C. 20525  
Attention: Dr. R. Roberts (1)

Headquarters

Wright Patterson AFB, Ohio 45433  
Attention: MAMP (1)  
MATB/G. A. Gegel (1)  
MAAM/Technical Library (1)  
AFSC-FTDS (1)  
AFML: MAM (1)  
MAG/Directorate of  
Materials (1)  
MAMP/I. Perlmutter (1)

Oak Ridge National Laboratory  
Oak Ridge, Tennessee 37830  
Attention: Technical Reports  
Library (1)

U. S. Atomic Energy Commission  
Washington, D. C. 20545  
Attention: Technical Reports  
Library (1)

U. S. Army Aviation Materials  
Laboratory  
Port Eustis, Virginia 23604  
Attention: SMOFE-APG/John White,  
Chief (1)

Army Materials Research Agency  
Watertown Arsenal  
Watertown, Massachusetts 02172  
Attention: S. V. Arnold, Director (1)

Advanced Metals Research Corporation  
149 Middlesex Turnpike  
Burlington, Massachusetts 01804  
Attention: Mr. J. T. Norton (1)

Aerospace Corporation (1)  
Reports Acquisitions  
P. O. Box 95085  
Los Angeles, California 90045

Allegheny Ludlum Steel Corporation  
Research Center  
Alabama and Pacific Avenues  
Brackenridge, Pennsylvania 15014  
Attention: Mr. R. A. Lula (1)

American Machine and Foundry  
1021 N. Royal Street  
Alexandria, Virginia 22314  
Attention: Dr. C. L. Morrison (1)

American Society for Metals  
Metala Park  
Novelty, Ohio 44073  
Attention: Dr. Taylor Lyman (1)

Avco Space Systems Division  
Lowell Industrial Park  
Lowell, Massachusetts 01851  
Attention: Library (1)

Commonwealth Scientific Corporation (1)  
500 Pendleton Street  
Alexandria, Virginia 22314

Battelle Memorial Institute  
505 King Avenue  
Columbus, Ohio 43201  
Attention: Dr. R. I. Jaffe (1)  
Dr. J. M. Blocher (1)  
Defense Metals Information  
Center (DMIC) (1)  
Dr. B. Wilcox (1)

The Bendix Corporation  
Research Laboratories Division  
Southfield, Michigan 48075  
Attention: Mr. E. C. Johnson (1)

Boeing Company  
P. O. Box 733  
Renton, Washington 98055  
Attention: W. E. Binz, SST Unit Chief (1)

Brush Beryllium Corporation  
17876 St. Clair Avenue  
Cleveland, Ohio 44112  
Attention: Mr. W. W. Beaver (1)

Case Institute of Technology  
University Circle  
Cleveland, Ohio 44106  
Attention: Professor L. Leonard (1)

Chromalloy Corporation  
Sintercast Division  
169 Western Highway  
West Nyack, New York 10994  
Attention: Mr. L. Maisel (1)

Curtiss-Wright Corporation  
Metals Processing Division  
760 Northland Avenue  
Buffalo, New York 14215  
Attention: Mr. B. Triffleman (1)

Denver Research Institute  
University Park  
Denver, Colorado 80210  
Attention: Library (1)

Douglas Aircraft Co. MFS  
3000 Ocean Park Blvd.  
Santa Monica, California 90406  
Attention: A-260/Dr. D. H. Killpatrick (1)

Fansteel Metallurgical Corporation  
Number One Tantalum Place  
North Chicago, Illinois 60064  
Attention: Library (1)

Ford Motor Company  
Materials Development Department  
20000 Rotunda Drive  
P. O. Box 2053  
Dearborn, Michigan 48123  
Attention: Mr. Y. P. Telang (1)

Firth Sterling, Inc. (1)  
Powder Metals Research  
P. O. Box 71  
Pittsburgh, Pennsylvania 15230

General Electric Company  
Materials and Processes Laboratory  
Schenectady, New York 12305  
Attention: Mr. C. T. Sims, Bldg. 55 (1)

General Electric Company  
Advanced Technology Laboratory  
Schenectady, New York 12305  
Attention: Library (1)

General Electric Company  
Materials Dev. Lab. Oper.  
Advance Engine and Tech. Department  
Cincinnati, Ohio 45215  
Attention: Mr. L. P. Jahnke (1)

General Motors Corporation  
Allison Division  
Indianapolis, Indiana 46206  
Attention: D. K. Hanink, Materials  
Lab. (1)

General Technologies Corporation  
708 North West Street  
Alexandria, Virginia 22314  
Attention: Library (1)

E. I. DuPont de Nemours and Co., Inc.  
Pigments Dept. Metal Products  
Wilmington, Delaware 19898  
Attention: Dr. Warren I. Pollock (1)

IIT Research Institute  
Technology Center  
Chicago, Illinois 60616  
Attention: Mr. N. M. Parikh (1)  
Mr. S. L. Blum (1)

Ilikon Corporation  
Natick Industrial Center  
Natick, Massachusetts  
Attention: Library (1)

International Nickel Company  
67 Wall Street  
New York, New York 10005  
Attention: Mr. R. R. Dewitt (1)

International Nickel Company  
P. D. Merica Research Lab.  
Sterling Forest  
Suffern, New York 10901  
Attention: Dr. F. Decker (1)

Ladish Company  
Government Relations Division  
Cudahy, Wisconsin 53110  
Attention: Mr. C. Burley, Jr. (1)

Arthur D. Little, Inc.  
20 Acorn Park  
Cambridge, Massachusetts  
Attention: Dr. B. Bovarnik (1)

Lockheed-Georgia Company  
Research Laboratory  
Marietta, Georgia 30060  
Attention: Dr. W. S. Cremens (1)

Lockhead Palo Alto Research Labs.  
Materials and Science Lab. 52-30  
3251 Hanover Street  
Palo Alto, California 93404  
Attention: Technical Information  
Center (1)  
Dr. C. G. Goetzel (1)  
Mr. E. C. Burke (1)

P. R. Mallory and Company, Inc.  
3029 E. Washington Street  
Indianapolis, Indiana 46206  
Attention: Technical Library (1)

Massachusetts Institute of Technology  
Metallurgy Department/RM 8-305  
Cambridge, Massachusetts 02139  
Attention: Prof. N. J. Grant (1)

Melpar, Inc. (1)  
3000 Arlington Boulevard  
Falls Church, Virginia 22903

Mitron Corporation (1)  
899 Main Street  
Waltham, Massachusetts 02154

Narmco Research and Development Div.  
Whittacker Corporation  
3540 Aero Court  
San Diego, California 92123  
Attention: Dr. F. J. Riel,  
Tech. Dir. (1)

N.R.A. Inc.  
35-01 Queens Boulevard  
Long Island City, New York 11101  
Attention: Dr. S. Grand (1)

North Star Research and Development  
Institute  
3100 Thirty-Eight Avenue, South  
Minneapolis, Minnesota 55406  
Attention: Dr. J. W. Clegg (1)

Nuclear Materials Company  
West Concord, Massachusetts 01781  
Attention: Dr. H. Levingston (1)

Ohio State University  
Columbus, Ohio 43210  
Attention: Prof. M. G. Fontana, Chairman  
Dept. of Metallurgical Eng. (1)

Rensselaer Polytechnic Institute  
Troy, New York 12180  
Attention: Prof. Fritz V. Lenel (1)  
Prof. G. S. Ansell (1)

Sherritt Gordon Mines, Ltd.  
Research and Development Division  
Fort Saskatchewan, Alberta, Canada  
Attention: Dr. D. J. I. Evans (1)

Solar Division  
International Harvester Corporation  
San Diego, California 92112  
Attention: J. V. Long, Director  
of Research (1)

Stanford Research Institute  
Menlo Park, California  
Attention: Mr. F. A. Halden (1)

Stanford University  
Palo Alto, California 94305  
Attention: Prof. Oleg Shergy (1)  
Dept. of Material Science

Sylvania Electric Products, Inc.  
Chemical and Metallurgical Division  
Towanda, Pennsylvania 18848  
Attention: Dr. J. S. Smith (1)

Texas Instruments, Inc.  
Materials and Controls Division  
P. O. Box 5474  
Dallas, Texas 75222  
Attention: Dr. Gene Wakefield (1)

TRW Electromechanical Division  
TRW Inc.  
23555 Euclid Avenue  
Cleveland, Ohio 44117  
Attention: Dr. A. S. Nemy (1)

Union Carbide Corporation  
Stellite Division  
Technology Department  
Kokomo, Indiana 46901  
Attention: Technical Library (1)

United Aircraft Corporation  
400 Main Street  
East Hartford, Connecticut 06108  
Attention: Research Library (1)  
E. F. Bradley, Chief (1)  
Materials Engineering

United Aircraft Corporation  
Pratt and Whitney Division  
West Palm Beach, Florida 33402  
Attention: Mr. J. Moore (1)

Universal-Cyclops Steel Corporation  
Bridgeville, Pennsylvania 15017  
Attention: Mr. C. P. Mueller (1)

Vitro Laboratories  
200 Pleasant Valley Way  
West Orange, New Jersey 07052  
Attention: Dr. H. McCullough (1)

Wah Chang Corporation  
Albany, Oregon 97321  
Attention: Mr. S. Worster (1)

Westinghouse Electric Corporation (1)  
MacArthur Avenue  
Bloomfield, New Jersey 07003

Westinghouse Electric Corporation  
Westinghouse Astronuclear Lab.  
P. O. Box 10864  
Pittsburgh, Pennsylvania 15236  
Attention: Mr. R. Begley (1)